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RESOURCE CONSERVATION AND RECOVERY ACT FACILITY INVESTIGATION FINAL COMPREHENSIVE QUALITY ASSURANCE MANUALS VOLUME V BOOK 3 OF 3 PAGE CHANGES REVISION 1 CNC CHARLESTON SC
7/30/1996
ENSAFE

COMPREHENSIVE LONG-TERM
ENVIRONMENTAL ACTION NAVY
NAVAL BASE CHARLESTON
CHARLESTON, SOUTH CAROLINA
CTO-029

FINAL
COMPREHENSIVE QUALITY ASSURANCE MANUALS
RCRA FACILITY INVESTIGATION
PAGE CHANGES, REVISION NO: 01

Prepared for:

DEPARTMENT OF THE NAVY
SOUTHERN DIVISION
NAVAL FACILITIES ENGINEERING COMMAND
CHARLESTON, SOUTH CAROLINA

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Release of this document requires the prior notification of the Commanding Officer of the Naval Base Charleston, Charleston, South Carolina.

VOLUME V
BOOK 3 of 3

LABORATORY
QUALITY ASSURANCE
PLAN

PACE, Inc. MN

Submitted by:

_____

Approved by:

Rodney T. Miller

Rodney T. Miller
Corporate Quality Assurance Officer

Rev. #0 - DATE: November 1, 1989
Rev. #1 - DATE: May 17, 1990
Rev. #2 - DATE: August, 1991

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III. INTRODUCTION, PROGRAM OBJECTIVES, AND STATEMENT OF POLICY

A. INTRODUCTION

This Generic Quality Assurance (QA) Plan is written in compliance with the elements required in the U.S. EPA, "Guidelines and Specifications for Preparing Quality Assurance Program Plans." (QAMS-004 80, September 20, 1980). This document contains the required elements of a Quality Assurance Plan and is prepared in such a way that entire sections can be referenced in subsequent specific project plans. This Laboratory QA Manual defines the systems of quality control and quality assessment that constitute the comprehensive Quality Assurance Program at PACE, Inc. Quality Control consists of specific procedures applied to all phases of analysis from sample receipt through the final reporting of results. The purpose of quality control is to insure that quality goals are met under routine operating procedures. Quality assessment involves the continuous evaluation of data and monitoring of analytical processes for the purpose of insuring that the quality control systems are performing effectively.

B. PROGRAM OBJECTIVES

The major elements of the overall Quality Assurance Program are summarized below:

- Use of appropriate methodologies by technically competent, well-trained personnel with state-of-the-art instrumentation and equipment.
- Adherence to well-defined standard operating procedures with emphasis on good laboratory and measurement practices.
- Analysis and assessment of quality control samples including (but not limited to) matrix spike samples, duplicate samples, surrogate spikes, blanks, and independent laboratory control standards.
- Participation in external quality evaluation programs such as the EPA Water Pollution and Water Supply (WP & WS) Study Programs.
- Maintainance of accreditation by State, Federal, and other applicable agencies for work performed.
- Monitoring of internal and external compliance to procedures and assessment of the performance of the analytical methods.

REFERENCES

1. Handbook for Analytical Quality Control in Water and Wastewater Laboratories, U.S. EPA 600/4-79-019, March, 1979.

2. Federal Register, 40 CFR Part 136, October 26, 1984.

3. Test Methods for Evaluating Solid Waste, U.S. EPA SW-846, September, 1986.

4. Quality Assurance of Chemical Measurements, Taylor, John K.; Lewis Publishers, Inc. 1987.

5. Standard Methods for the Examination of Water and Wastewater, APHA, AWWA, WPCF: 16th Edition, 1985.

6. NIOSH Manual of Analytical Methods, U.S. Department of Health, Education, and Welfare; Second Edition, 1977.

7. Methods for Non-conventional Pesticides Chemicals Analysis of Industrial and Municipal Wastewater, Test Methods, EPA-440/1-83/079-C.

8. Methods for Chemical Analysis of Water, Wastes, EPA-600/4-79--020, 1983.

9. California Administration Code, Title 2, Chapter 30, Article II, "Criteria for Identification of Hazardous and Extremely Hazardous Wastes."

10. The Determination of Inorganic Anions in Water by Ion Chromotography - Method 300.0 Test Method, EPA-600/4-84-017. March, 1984.

11. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846, 2nd Edition, U.S. EPA, revised April, 1984.

XVI. QUALITY ASSURANCE REPORTS TO MANAGEMENT

Quarterly reports are provided by the Quality Assurance officer to the President, Vice President of Quality and Regional Director. This report addresses the quarterly quality assurance activities including details of corrective actions implemented, audit results, and QC summary information.

XV. CORRECTIVE ACTION

If, as a result of audits or QC sample analyses, methods systems prove to be unsatisfactory, corrective action shall be implemented. The project manager, department manager, Quality Assurance Officer, supervisor, and analyst may be involved in the corrective action. If previously reported data are affected by a situation requiring correction or if the corrective action impacts a project budget or schedule, the action will directly involve the project manager (and Quality Assurance Officer).

For immediate or long-term corrective actions, steps comprising a closed-loop corrective action system are as follows:

1. Define the problem.

2. Assign responsibilities for problem investigation.

3. Investigate and determine the cause of the problem.

   a. Check all calculations

   b. Re-analyze the sample

   c. Verify the integrity of the spiking solution, laboratory control sample, or calibration standard.

   d. Check instrument and operating conditions to preclude the possibility of malfunctions or operator error.

4. Determine the corrective action(s) necessary to eliminate the problem.

5. Assign and accept responsibilities for implementing the corrective action.

6. Establish the effectiveness of the corrective action and implement the correction.

7. Verify and document that the corrective action has eliminated the problem (using a Discrepancy Report form)

   Depending upon the nature of a problem, the corrective action implemented may be formal or informal. In either case, occurrence of the problem, the corrective action employed, and verification that the problem has been eliminated must be documented.

   In addition, if the corrective action mandates the preparation of a new standard or calibration solution(s), a comparison study between the new solution versus the old solution will be performed. The results are supplied with the weekly QC submittal as verification of problem elimination.

E. COMPLETENESS

Data completeness can be quantified during data assessment. It is expected that laboratories should provide data, meeting QC acceptance criteria, for 95% or more of the requested determinations. It is incumbent for planners to identify any sample types, such as control or background locations, which require 100% completeness.

F. REPRESENTATIVENESS

Representativeness is a qualitative element that is related to the ability to collect a sample that reflects the characteristics of that part of the environment that is to be assessed. Sample representativeness is dependent on the sampling techniques used and is considered individually for each project. It is specifically addressed in each work plan.

G. COMPARABILITY

Comparability is also considered during preparation of the work plan. The objective of comparability is to ensure that results of similar activities conducted by different parties are comparable. For example, the use of EPA-approved, etc., methods and procedures ensure comparability with other data from previou following studies.

D. CONTROL LIMITS

Control limits represent the 99% confidence interval and are equal to the mean value of the control sample, plus or minus three standard deviations (±3S). Exceeding these limits indicates that the analytical system is out-of-control. The Quality Assurance Officer or the supervisor shall be informed and corrective action shall be taken.

1. Method of Setting Limits

Control limits are established via statistical analysis, using QC sample results. Limits are determined for a parameter of each method as analyzed on a specific instrument.

The mean value (P) and the standard deviation (S) for each data set is calculated and the limits are set as:

Warning (WL) = P + 2S = 95% Confidence limit
Control (CL) = P + 3S = 99% Confidence limit

Where $P = \frac{X1 + X2 + X3 \ldots Xn}{n}$ x = Sample result

and $S = \frac{\sum(X - P)^2}{n-1}$ n = Total # of results in set
P = mean value

The minimum number of results to be used for statistical calculation (n) is 15-20. Limits will generally be calculated from a data point set every 30 days, depending on the method. Updated limits are issued at the beginning of every month.

2. Utilization of Acceptance Limits

QC sample results must fall within the established warning limits (P ± 2S) for each parameter.

Results that fall outside of warning limits, but remain within the control limits (P ± 3S), are consider suspect. These results must be carefully examined for possible sources of error in the analysis, or justified as a matrix bias effect. All such results are recorded in a Discrepancy Report form/Corrective Action form (See section XV).

Any three consecutive results outside of warning limits but within control limits is an out-of-control event which shall be documented and corrected.

Results that fall outside of control limits (P ± 3S) must be documented and corrective action taken.

Six consecutive points on the same side of an established mean indicate a trend and require corrective action.

EXHIBIT 26

NO3-NICL.XLS

PACE, Inc.
QUALITY ASSURANCE
PRECISION CONTROL CHART

Matrix: Water

DATE: ########
TIME: 14:35:37

Compound: [illegible]

Method: EPA#300.0

Low=1-100mg/l

MDL=1mg/l

	Date	DUPLICATES X1	X2	Relative Percent Difference (RPD)	LCL Ave-3sd (Min=0)	LWL Ave-2sd (Min=0)	Average RPD	UWL Ave+2sd	UCL Ave+3sd
2	2/22/91	1.10	1.19	[illegible]	0	0	2	8	11
3	3/6/91	0.80	0.80	[illegible]	0	0	2	8	11
4	3/7/91	0.98	0.99	[illegible]	0	0	2	8	11
5	3/13/91	1.05	1.05	[illegible]	0	0	2	8	11
6	4/21/91	3.50	3.50	[illegible]	0	0	2	8	11
7	4/24/91	10.60	10.60	[illegible]	0	0	2	8	11
8	4/27/91	2.10	2.10	[illegible]	0	0	2	8	11
9	4/28/91	1.40	1.30	[illegible]	0	0	2	8	11
10	5/3/91	4.10	4.00	[illegible]	0	0	2	8	11
11	5/13/91	9.30	9.10	[illegible]	0	0	2	8	11
12					0	0	2	8	11
13					0	0	2	8	11
14					0	0	2	8	11
15					0	0	2	8	11
16					0	0	2	8	11
17					0	0	2	8	11
18					0	0	2	8	11
19					0	0	2	8	11
20					0	0	2	8	11
1					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11
					0	0	2	8	11

Average RPD	[illegible]
Std Deviation	[illegible]

BENZ-DS.XLS

PACE , Inc.
QUALITY ASSURANCE
SPIKE RECOVERY CHART

Instrume D
Matrix: Groundwater

DATE: 00000000
TIME: 14:17:40

Compound: [redacted]
Method: EPA0602

	Date	ACTUAL Spike Recovery	THEORY Spike Recovery	[redacted]	LCL Ave-3sd	LWL Ave-2sd	Average Recovery	UWL Ave+2sd	UCL Ave+3sd
2	4/5/91	19.00	20.00		67	81	110	138	152
3	4/5/91	45.00	40.00		67	81	110	138	152
4	4/5/91	20.00	20.00		67	81	110	138	152
5	4/6/91	23.00	20.00		67	81	110	138	152
6	4/7/91	24.00	20.00		67	81	110	138	152
7	4/8/91	23.00	20.00		67	81	110	138	152
8	4/8/91	53.00	40.00		67	81	110	138	152
9	4/9/91	16.80	20.00		67	81	110	138	152
10	4/11/91	23.00	20.00		67	81	110	138	152
11	4/12/91	29.00	40.00		67	81	110	138	152
12	4/18/91	23.00	20.00		67	81	110	138	152
13	4/18/91	43.00	40.00		67	81	110	138	152
14	4/24/91	50.00	40.00		67	81	110	138	152
15	4/25/91	41.00	40.00		67	81	110	138	152
16	4/25/91	24.00	20.00		67	81	110	138	152
17	4/26/91	45.00	40.00		67	81	110	138	152
18	4/29/91	24.00	20.00		67	81	110	138	152
19	4/30/91	20.00	20.00		67	81	110	138	152
20	5/1/91	48.00	40.00		67	81	110	138	152
1	5/2/91	43.00	40.00		67	81	110	138	152
					67	81	110	138	152
					67	81	110	138	152
					67	81	110	138	152
					67	81	110	138	152
					67	81	110	138	152
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					67	81	110	138	152
					67	81	110	138	152
					67	81	110	138	152
					67	81	110	138	152
					67	81	110	138	152
					67	81	110	138	152

Ave Recovery	[redacted]
Std Deviation	

EXHIBIT 24

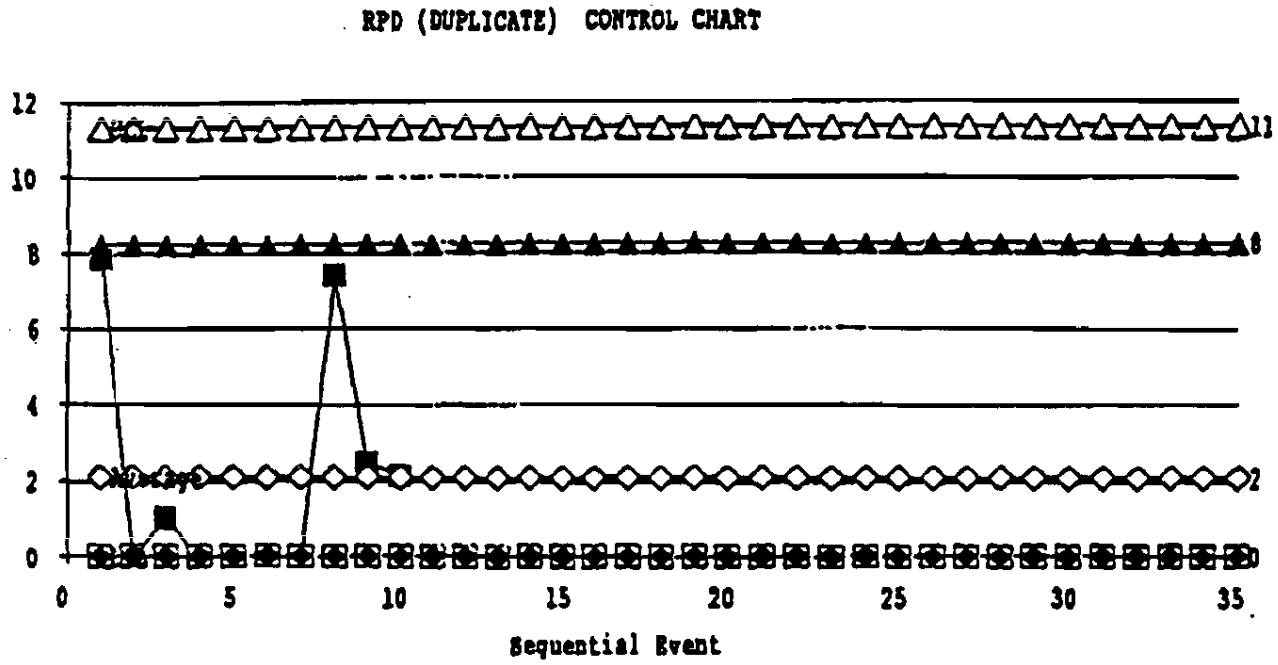
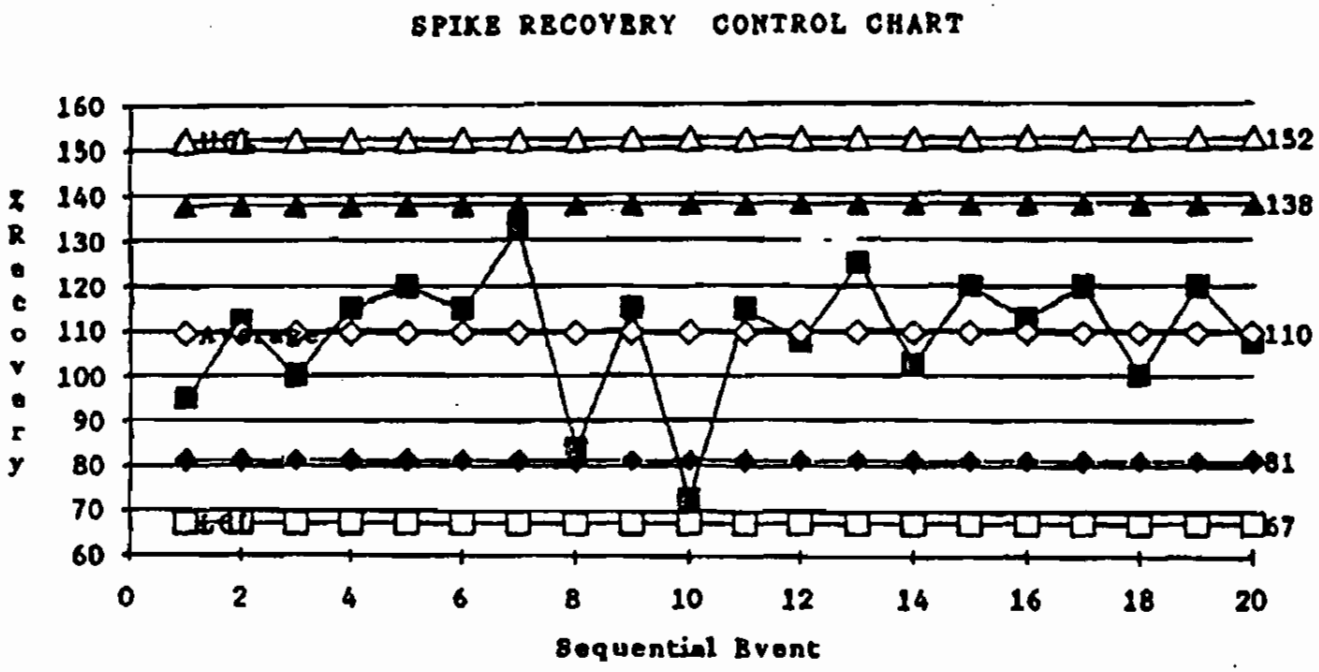

RPD (DUPLICATE) CONTROL CHART
12
10
8
6
4
2
0
11
8
2
0
0
5
10
15
20
25
30
35
Sequential Event

EXHIBIT 23


SPIKE RECOVERY CONTROL CHART
% Recovery
160
150
140
130
120
110
100
90
80
70
60
UCL
Average
LCL
152
138
110
81
67
0
2
4
6
8
10
12
14
16
18
20
Sequential Event

XIV ASSESSMENT OF PRECISION, ACCURACY, COMPLETENESS REPRESENTATIVENESS, AND COMPARABILITY

The Quality Control Program at PACE uses precision and accuracy data to determine the acceptability of analytical results. Precision refers to result reproductibility and accuracy measures the degree of difference between observed and true values. One of every 20 analyses performed at PACE is run in duplicate (precision). Also, one of every 20 samples is spiked with a standard to assist in evaluating the accuracy of the method. Once 20 sets of precision or accuracy data have been obtained, a quality control chart is prepared. The Shewhart technique is the statistical method used to construct the charts. These quality control charts provide a quick visual means for monitoring the daily performance of the laboratory. Exhibits 23 and 24 contain generic examples of accuracy and precision charts along with their corresponding data sheets (Exhibits 25 and 26).

A. ACCURACY

The actual test result is compared to the theoretical result of 100% recovery and the percent recovery is calculated.

$$\% \text{ Recovery} = \frac{\text{Spiked Sample Result} - \text{Sample Result}}{\text{Spike Quantity}} \times 100$$

The percent recovery must fall within specific control limits for the results to be accepted and subsequent data validated. (See Table 2)

B. PRECISION

The results of the duplicate analyses are computed and the absolute relative percent difference (RPD) is calculated.

$$\text{RPD} = \frac{|\text{Sample Result} - \text{Duplicate Result}|}{\text{Average Result}} \times 100$$

The RPD must fall within set control limits for the results to be accepted and subsequent data validated. A one-sided distribution with zero as a target value is typical, given absolute value requirements (CLP).

C. WARNING LIMITS

Warning limits represent the 95% confidence interval and are equal to the mean value for the control sample, plus or minus two standard deviations (± 2S). Exceeding these limits is a warning that the analytical system may be approaching an out-of-control situation, and should be inspected for possible sources of error before continuing the analysis. Analysts will inform the Quality Assurance Officer or the supervisor of such problems.

EXHIBIT 22

INSTRUMENT MAINTENANCE LOGBOOK FORM

INSTRUMENT I.D.:

DATE	MAINTENANCE ACTIVITY	NAME

XIII. PREVENTIVE MAINTENANCE

PACE maintains service contracts for most major analytical equipment including all chromatography instruments, balances, atomic absorption, and inductively coupled plasma instruments. All instruments and equipment receive routine preventive maintenance, which is recorded in instrument specific maintenance logs. Routine maintenance insures that all equipment is operating under optimum conditions, reducing the possibility of instrument malfunction (consequently affecting sample results). An example of an instrument maintenance log is included (Exhibit 22).

B. TRAINING AND TECHNICAL REVIEW

PACE considers competent, well-trained personnel to be a key to successful production of valid and reliable data. An extensive training and technical review program is in place at PACE, Inc. It includes:

1. Training Plans

The type of training required for each new or transferred employee is determined individually. A training plan is established to reflect general training needs and to fulfill job requirements.

2. Training Classes

All sections conduct regularly scheduled training sessions specific to their needs.

Audio/visual training programs and open learning texts are available for use by all personnel.

Other laboratory QA and general training classes are offered periodically.

3. Technical Review Program

All employees are subject to technical reviews with their supervisor. The technical review assesses an individual's training progress and technical development and provides an opportunity to redirect the training plan accordingly to comprehensively cover further developmental needs. The schedule for technical reviews is:

a. New hire or transfer to new position/responsibilities: 6 months, 1 year.

b. After 1 year in same position/responsibilities: annually.

4. Support Programs

Attendance at outside seminars, classes, etc., is highly encouraged. PACE participates in many of these throughout the year. In-house seminars are presented by employees for employee bi-monthly meetings. Various topics are covered, including regulatory items and information from attendance at outside seminars. The PACE in-house library contains current periodicals and journals pertinent to the environmental industry and analytical chemistry, in addition to reference books, text books, and regulatory publications.

Blank Analyses:

Frequency -
Documentation -
Criteria -

Quality Control Chart - _____ Yes _____ No Type _____

Latest Entry - _____

Log Book Available - _____ Yes _____ No Maintenance Record _____

Type of Records -
Latest Entry -

External Standards Used - _____ Yes _____ No

Type -
Origin -
Traceability -

Participation in External P.E. Program:

Program -
Frequency -
Latest Participation -
Results Available -

Evaluation of this Analysis Done During On-site Audit -

_____ Yes _____ No Agency: _____

Latest Evaluation -

Describe (in detail) the Data Review Process that is used for QC and sample analytical data. Especially describe the role of analyst, supervisor, manager, director, QAO.

_____
_____
_____
_____
_____
_____
_____
_____
_____
_____

Documented - _____ Yes _____ No

Information Provided By: _____
Auditor's Signature _____ Date _____

Page 2
Audit Information

ADHERANCE TO SOP:

Without Exception - _____ Yes _____ No - if no, describe specific variance(s) in detail:

_____

_____

_____

QUALITY CONTROL:

Calibration Protocol -

General Procedure (i.e., 3 pt, 5 pt.) _____

_____

_____

Specific

Initial (new) Calibration Procedure - _____

_____

Frequency -

Last Documented New Calibration -

Continuing Calibration Procedure -

Frequency -

Last Documented Continuing Calibration -

EPA Check Standard used - _____ Yes _____ No Frequency _____

Last Documented Use -

Spike Recovery Data:

Frequency -
Documentation -
Criteria -

Duplicates:

Frequency -
Documentation -
Criteria

EXHIBIT 21

AUDIT INFORMATION
ANALYTICAL OPERATIONS

ANALYTICAL INFORMATION:

Analysis -

Method(s) -

Analyst(s) -

Instrument(s) -

CERTIFICATION INFORMATION:

_____ Yes _____ No (If yes, name of agency) _____

STAFF TRAINING:

Type -

Documentation -

STANDARD OPERATING PROCEDURES:

Hard Copy - _____ Yes _____ No PACE Format - _____ Yes _____ No

Latest Revision Date -

Utilized (how long) -

Copy Provided to Corporate Quality - _____ Yes _____ No

MDL STUDY: _____ Yes _____ No (if yes, provide date) _____

Protocol Used - EPA _____ Other _____(specify)

Documentation - _____ Yes _____ No - Available in lab _____ Filed _____

Approved by - _____ Analyst: Name(s) and Title(s) _____ Date

_____ Quality Assurance Office: _____

pace
INCORPORATED
THE ASSURANCE OF QUALITY

EXHIBIT 20

National Certifications

July 1991

Certifications/ Accreditations	Regional Office: NC Ashe	NC Char	CO	FL	IA	KS	MN	NY	No CA	PA	So CA
Successful Participation in U.S.EPA Contract Laboratory Program (CLP)						●	●	●			
U.S.Army Toxic and Hazardous Materials Agency (USATHAMA)							●				
DOE Hazardous Waste Remediation Action Program (HAZWRAP)							●	●			
Naval Energy and Environmental Support Activities (NEESA)							●	●	●		
American Industrial Hygiene Association (AIHA) Laboratory		●	●				●				
National Voluntary Laboratory Accreditation Program (NVLAP)			●	●			●				
Audited by the Missouri River Division of the U.S. Army COE				○			●		●	●	
NIOSH Proficiency in Analytical Testing (PAT) Program		●	●				●				
Analytical Support Laboratory for Minnesota Superfund Projects							●				
Nuclear Materials License			●								

● Certified ○ Certification Pending

pace INCORPORATED
THE ASSURANCE OF QUALITY

EXHIBIT 19

State Certifications

July 1991

State Certification	Regional Office: NC Ashe	NC Char	CO	FL	IA	KS	MN	NY	No CA	PA	So CA
AL Drinking Water				●							
CA Air							●		●		
CA Pesticides									●		
CA Hazardous Waste						○	●		●		
CA Wastewater						○			●		
CA Drinking Water						○			●		
CO Drinking Water			●			○					
CT Solid and Hazardous Waste								●			
CT Drinking Water								●			
CT Wastewater								●			
CT Environmental										●	
DE Drinking Water										●	
FL Environmental				●			●	○			
FL Drinking Water				●							
IA Drinking Water				●	○	○					
IL Drinking Water							○				
IN Drinking Water										●	
KS Drinking Water						●	●	●			
KS Solid and Hazardous Waste						●	●	●			
KS Environmental							●				
KS Wastewater								●			
KY Drinking Water				●							
MA Drinking Water								●			
MD Drinking Water								○		○	
MI Drinking Water							●				
MN Drinking Water (Microbiological)							●				
NC Biotoxicity	●										
NC Drinking Water	●	●		●							
NC Wastewater	●	●		●							
ND Drinking Water						●	●				
NJ Drinking Water								●		●	
NJ Wastewater								●		●	
NY Air								●			
NY Drinking Water							●	●			
NY Solid and Hazardous Waste							●	●			
NY Wastewater							●	●			
PA Drinking Water										●	
RI Drinking Water										●	
RI Solid and Hazardous Waste										●	
RI Wastewater										●	
SC Drinking Water	●	●									
SC Environmental		●		●							
TN UST				●							
TN Drinking Water		●									
VA Drinking Water		●									
VA Wastewater		●									
WI Drinking Water							●				
WI Environmental							●				
WV Drinking Water										●	

● Certified ○ Certification Pending

XII. PERFORMANCE EVALUATIONS AND SYSTEM AUDITS

A. PACE's SYSTEM AUDITS

Internal Audits:

1. All records, logs, and data files are audited quarterly for completeness, accuracy, and adherence to standard operating procedures by an internal auditing team. Audit team members include the Quality Assurance Officer and any other associated personnel. Several random project files are evaluated for compliance to procedures throughout the analytical process (i.e., from sample receipt through the final report). Supervisors, and lab analysts routinely check all records for the same criteria.

External Audits:

2. PACE is audited as required by regulatory agencies to maintain laboratory certifications, and by various commercial clients with laboratory auditing programs. These audits include audits by USEPA, USATHAMA, AIHA, and other appropriate federal, state and private agencies.

Total Quality System Audit:

3. The Corporate Quality Office performs a yearly on-site audit at each regional facility. The Corporate audit is conducted by the Vice President of Corporate Quality/the Quality Program Specialist. This audit is designed to evaluate all regional office operations and is not limited to only laboratory operations. Audits may either be systems-related or technical in nature, depending on the type of information needed for making quality improvements. An example of one type of form used is shown in Exhibit 21.

B. PERFORMANCE EVALUATIONS:

1. PACE participates in the US EPA semi-annual drinking water (WS Series) and semi-annual wastewater (WP Series) Performance Evaluation Studies (four studies per year).

2. PACE participates in various client-sponsored performance evaluations by analyzing QC samples prepared and submitted by commercial clients in conjunction with their own QA program.

3. Several government proficiency samples are analyzed annually to maintain various laboratory certifications (Exhibit 19 and 20).

4. PACE regional offices are provided blind QC check samples quarterly. These are provided by Corporate Quality as a part of the PACE Interregional Testing Survey, and may also be provided independently by the regional Quality Assurance Officer.

For non-instrumental general (wet) chemistry methods, the MDL is established using titrimetric or gravimetric procedures. The MDL for titrimetric procedures is equal to the concentration calculated from a volume of titrant which is one-half of the smallest buret increment. Gravimetric MDLs are calculated from the weight equal to the lowest possible scale reading.

F. CONTROL CHARTS

Control charts monitor daily variations in precision and accuracy of routine analysis and detect variation trends. QC charts are constructed from performance data of the complete analytical method. Control chart construction requires initial data to establish the mean and standard deviation of measurements. Currently, spikes, spike duplicates, RPD's and external check sample values are charted.

G. LABORATORY CONTROL SAMPLES

NIST traceable quality control check samples are analyzed at least quarterly. They provide a means of assessing the accuracy and precision of a measurement system's performance. Parameters of interest that initially fall outside of QC acceptance criteria are compared against a prepared EPA QC check sample. If laboratory performance for the parameter is found to be out of control, then necessary corrective actions are implemented.

EXHIBIT 18

VERT STANDARDS:

NAME: Acephate CODE: Acephate-1

OTHER NAMES: Methanamidophos

BRAND: Chem Service WARNING: POISON

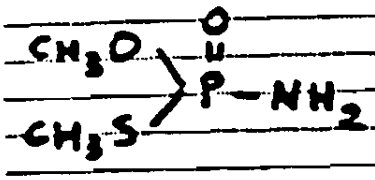
CAT. NO: PS-738

LOT NO: 30-13 LOCATION: Fr. # 2

PURITY: 98% Rack # 1

EXP. DATE: 9-90 position # 23

RECEIV. DATE:



Solvent used: Acetone
Source:
purity:
Lot #:

GC Extraction

DILUTIONS:

CODE: Acephate-D 1

STOCK SOLUTIONS AND STANDARDS

STD #	CONC. (PPM)	SOLVENT	PREP. DATE	LOCATION
# 502	2,000 ppm	Acetone	3-1-89	Fr. #1, Rack # 5
# 503	D.W. 80 ppm	Methanol	3-2-89	Fr. #1, Rack #7
# 503 A	40 ppm	"	"	"
# 503 B	60 ppm	"	"	"
# 503 C	100 ppm	"	"	"

GC Extraction

Exhibit 18 illustrates a standard log book entry. Standard Operating Procedure for standards preparation contains further instructions for assigning unique ID numbers, proper syringe technique, shelf life of standards, and good laboratory practices.

Labeling: The standard vial should have a reference label (covered with cellophane tape) with the following information:

1 - Standard
2 - Name of Standard
3 - Prep. Date
4 - Prep. Personnel Initials
5 - Solvent

Certified reference standards from the EPA Repository are used for calibration or laboratory control standards in many organic analyses. Reference standards may also be purchased from approved commercial vendors. Currently approved vendors for organic reference standards are Ultra-Scientific, Supelco, Chem-Service, Inc., and Aldrich Chemical Company, Inc. Inorganic standards are purchased from major scientific supply companies (Fisher, American Scientific, and VWR). Certificates of analyses are requested with each purchase.

E. METHOD DETECTION LIMIT

The method detection limit (MDL) is defined as the minimum substance concentration that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero. In general, the protocol described in Appendix B to 40CFR 136 (Federal Register, Vol. 49, No. 209, 10/26/84) is used to establish MDL's.

For GC/MS analyses and organochlorine pesticides by GC/EDC, the MDL has been determined according to EPA Contract Required Detection Limits (CRDL) as established for the Contract Laboratory Program. The MDL's for other organic analyses are set according to industry standards, client requirements, and instrument/method limitations. The MDL is validated using prepared standard solutions analyzed at detection limit concentrations.

Metals and general (wet) chemistry analyses MDL's correspond to instrument detection limits, and are established in the following manner: A standard solution of analyte in laboratory pure water with a concentration of 3-5 times the estimated instrument detection limit is analyzed seven consecutive times. The MDL is set at 3 times the standard deviation of the seven consecutive measurements.

B. MATRIX SPIKE AND SURROGATE ADDITIONS

Accuracy and matrix biases are monitored using spiked samples and where possible, surrogate additions. It is standard policy throughout the laboratory to prepare and analyze at least one matrix spike for each batch of 20 samples, for each matrix type within the batch, or as specified by state/project requirements.

Surrogate spiking compounds (if available), are added to and analyzed for, with every sample. A measured amount of spike/surrogate concentration is added to the sample before extraction or preparation. Surrogate spiking is utilized for GC and GC/MS analyses only.

C. DUPLICATE SAMPLE ANALYSIS

Precision is assessed by result comparison of a sample prepared and analyzed in duplicate. It is standard policy throughout the laboratory to prepare and analyze at least one duplicate sample for each batch of 20 samples and matrix type within the batch, or as specified by state/project requirements.

D. STANDARDS

The term standard shall apply to any analyte solution of known concentration which is traceable to a certified reference material. This includes calibration standards, spiking solutions, and laboratory control samples. Claims of traceability establish the accuracy of measurements. Therefore, maintaining standard traceability is critical to the achievement of known and defensible data quality.

To establish traceability, all purchased reference materials (neat and stock solutions) are recorded into section-specific standard log books when received.

All entries and PACE standard labels contain a unique PACE ID number, date received, date opened, and expiration date. Log book entries also include the manufacturer's lot number, certified purity, and storage location. Subsequent preparations of stock, intermediate, and working solutions are also recorded in the standard log books. These entries must include all discrete measurements made during a preparation, parent materials, solvent used, and a PACE ID number.

XI. INTERNAL QUALITY CONTROL

PACE, Inc. quality assurance practices consist of general quality control and assessment procedures that are adapted to the specific operating conditions within each section. The general elements of quality control are outlined below.

A. BLANK ANALYSIS

Reagent: A reagent blank consists of laboratory pure water and any reagents added to a sample during analysis only, or straight solvent.

Method blank: A method blank is a water or soil blank which undergoes all of the preparation procedures applied to a sample (i.e., extraction, digestion).

It is standard policy throughout the laboratory to prepare and analyze a reagent or method blank (whichever is appropriate) with each sample batch. Separate water and soil method blanks are prepared for mixed matrix batches.

Reagent blanks may also be inserted at regular intervals on large batches (of no more than 20 samples), or after highly concentrated samples to check for carryover/contamination. For methods utilizing surrogate compounds, the surrogates are added to all blanks and are subject to meeting acceptance criteria.

A trip blank is submitted for analysis with most samples analyzed for volatile organic compounds. A field blank or procedure blank may also be submitted at the discretion of the client. Field, procedure, and trip blanks are analyzed upon request of the client. Reagent blanks are run daily on each instrument to check the contaminant level.

INFORMATION FLOW SCHEMATIC

EXHIBIT 17

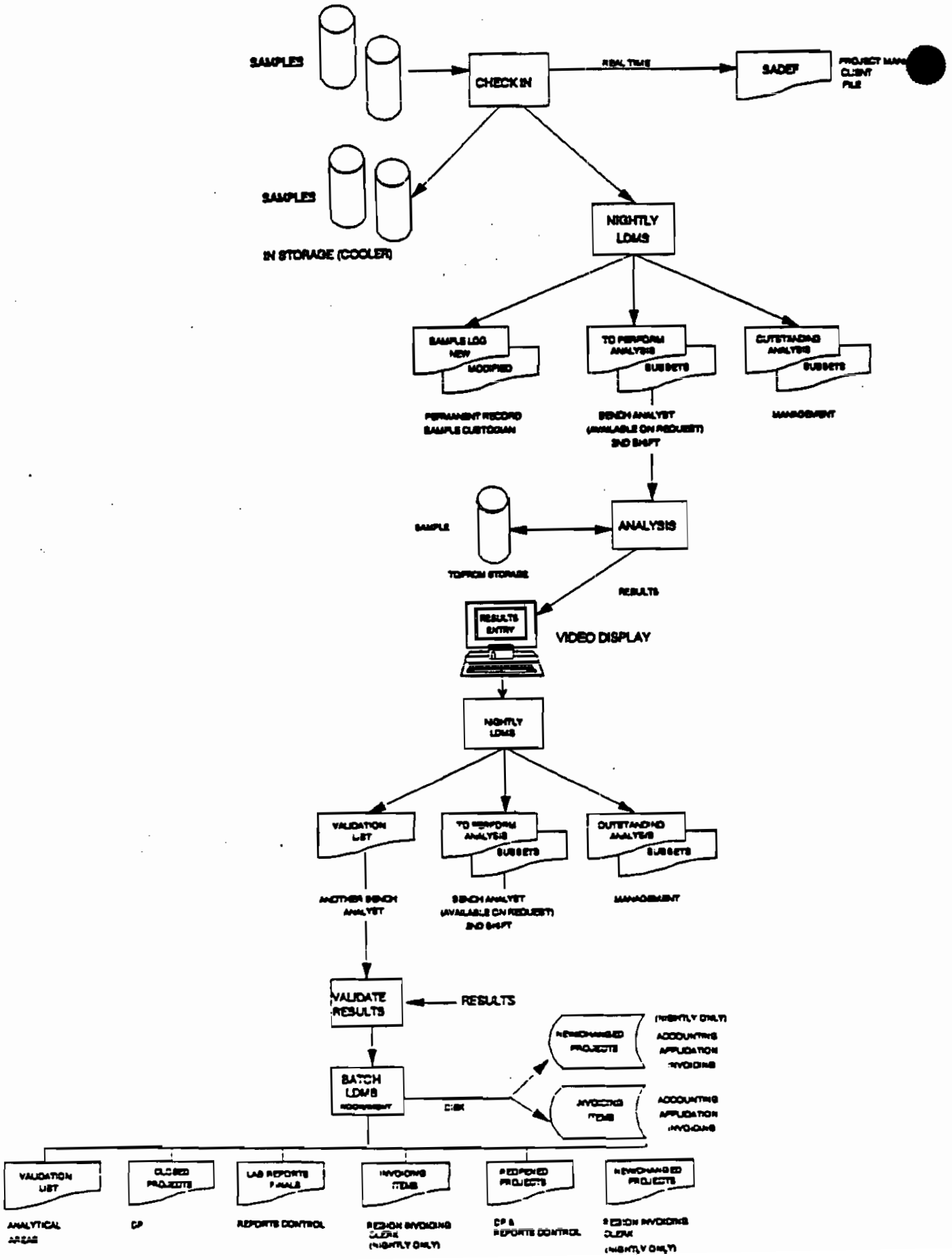
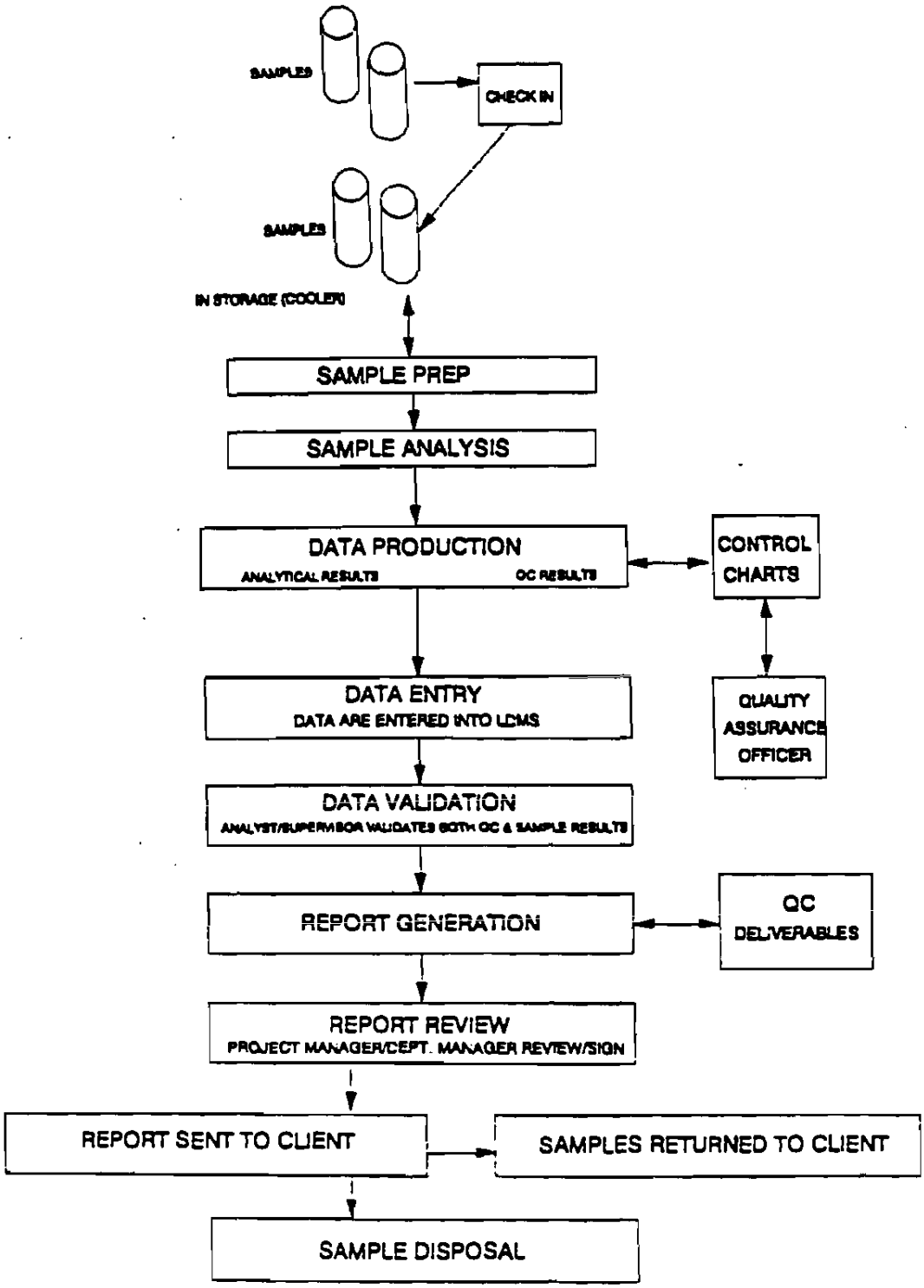


EXHIBIT 16

LABORATORY SAMPLE FLOW SCHEMATIC



X. DATA REDUCTION, VALIDATION AND REPORTING

Final results are entered into the LDMS system by the analyst, independently reviewed/validated by another analyst or supervisor experienced in the method, and approved by the department manager/lab director. Exhibit 16 describes the flow of samples through the laboratory.

All quality criteria (accuracy, precision, control limits, etc.) are reviewed and approved by the technical staff and independently monitored by the Quality office. Each project is assigned to a project manager. The project manager is responsible for tracking sample progress in the laboratory and ensuring delivery of the product as specified by the client.

The report is approved and signed by the department manager or director.

Complete project files are periodically inventoried and stored off-site in a secure facility. Electronic data are copied onto computer tape, inventoried and stored off-site in a secure facility.

Sample information flow through the LDMS, and the sequential generation of reports used to manage workloads are illustrated in Exhibit 17.

TABLE #3

ACCEPTANCE CRITERIA FOR QUALITY CONTROL SAMPLES & INSTRUMENT CALIBRATION

	MATRIX SPIKE % RECOVERY	SURROGATE SPIKE % RECOVERY	RPD DUPLICATE SAMPLES	INITIAL CALIBRATION LINEARITY	CALIBRATION VERIFICATION	LCS/EPA QC SAMPLE
GC	Within calculated control limits*	Within calculated control limits	≤ maximum RPD acceptance limit	RSD ≤ 20%	± 15% of true value or initial response	±15% of true value or EPA limit
MS	Within calculated control limits*	Within calculated control limits	≤ maximum RPD acceptance limit	RSD ≤ 30%	± 30% of initial average RF	± 15% of true value or EPA limit
GENERAL CHEMISTRY	Within calculated control limits*	N/A	0-67 on samples < 10x MDL 0-20 on samples > 10x MDL MDL = Method Detection Limit	Correlation coefficient ≥ .995	± 10% of true value	± 15% of true value or EPA limit
METALS	Within calculated control limits*	N/A	0-67 on samples < 10x MDL 0-20 on samples > 10x MDL MDL = Method Detection Limit	Correlation coefficient of: ≥ .995 ≥ .995 :AA ≥ .995 :Cold Vapor	± 10% of true value	± 15% of true value or EPA limit

Establisment and Utilization of Acceptance Limits

G. ACCEPTANCE CRITERIA AND CONTROL CHARTS

General acceptance criteria for quality control samples and instrument calibration/verification are summarized in Table 3. Internal in-house control limits are regionally generated for specific methodologies and instruments to achieve methodology and regulatory requirements.

PROJECT #

GC EXTRACTION

BATCH #

Location	Sample Number	Weight of Sample	Date & Time of Extraction	Final Volume	Date of Conc.	% Emulsion	Comments	Extraction Location	Column	NCLSON File	% Recovery	Date & Time

EXTRACTION METHOD

Separatory Funnel ☐

Continuous Liq/Liq ☐

Soxhlet ☐

Sonication ☐

Other: _____ ☐

Spike #

Dup. Spike #

QUALITY CONTROL INFORMATION

Surrogate:

Spike:

ROUTING

Person Who: Initial

Extracted _____

Concentrated _____

Supervisor _____

GC/MS _____

PROJECT #

GC-MS EXTRACTABLES

BATCH #

Sample Location	Sample Number	Date/Time of Extraction	Initial Volume	Surrogate	Spike	Final Volume	Date of Conc.	% Emulsion	Comments	Extract Location

EXTRACTION METHOD

- Separatory Funnel ☐
- Continuous Liq/Liq ☐
- Soxhlet ☐
- Sonication ☐
- Other: _____ ☐

Spike #

Dup. Spike #

QUALITY CONTROL INFORMATION

Surrogate:

Spike:

ROUTING

Person Who:	Initial
Extracted	_____
Concentrated	_____
Supervisor	_____
GC/MS	_____

NPPWGJ5

b. Reagent Blank

Any reagent blank result greater than the MDL terminates the analysis until corrective action resolves the problem. For ICP metals, a negative blank value greater than two times the MDL also requires corrective action. In rare cases, if all corrective action fails to resolve the problem, sample and blank data are reported if the problem cannot be rectified.

E. GENERAL CHEMISTRY PROCEDURES

1. Calibration and Verification

All instruments are calibrated daily with 3-6 point curves, depending upon instrument requirements. The calibration is continuously verified throughout the run, with either a calibration standard or laboratory control standard inserted after every 10th sample.

2. Laboratory Control Sample

A laboratory control sample is analyzed at least once during each batch of samples.

3. Matrix Spike and Duplicate Samples

Performed at a minimum of every 20 samples, or as specified by state/project requirements.

F. RECORD KEEPING AND REVIEW

All records and data are generally logged into hard cover bound books.

The extractions section utilizes method-specific bound books to record all data pertaining to sample extraction and preparation. An extraction benchsheet is used to transfer information to GC or GC/MS with each extracted sample or batch (Exhibit 14 and 15).

The organic and inorganic departments utilize benchsheets, maintained by analysts; specific for injection data and instrument maintenance. Spectras and chromatograms are filed by acquisition date.

The individual analysts and technicians are responsible for maintaining accurate, legible records and logs in accordance with standard operating procedures. The supervisors are responsible for ensuring adherence to procedures.

Secondary review of all records and logs is performed by someone other than the person generating the document, preferably the department supervisor. Evidence of secondary review is provided on the document as initials and review date by the secondary person.

See Section X for magnetic media storage.

7. **Reagent/Method Blank**

VOA - one per 12-hour per shift
BNA - one per batch of samples extracted

Common laboratory solvents present in the blank at a concentration less than 3 times the MDL will be footnoted on the analysis report. Common solvents at greater concentrations or the presence of any contaminant not considered a common laboratory solvent at a concentration greater than the MDL indicates the need to re-extract/re-analyze the blank and associated samples.

D. METALS PROCEDURES

1. Calibration and Calibration Verification

All instruments are calibrated at the start of each run. The Flame AA and graphite furnace requires a 4 point calibration. The ICP methods utilize a minimum of 4 points. Cold vapor analysis of mercury requires a 5 point calibration. Calibration verification is performed after 10 samples, or more often if indicated by the laboratory control sample. ICP uses calibration procedures as stated in the method or according to manufacturer's recommended calibration procedures.

2. Laboratory Control Sample

Performed at a minimum of every 20 samples, or as specified by state/project requirements.

3. Matrix Spike

Performed at a minimum of every 20 samples, or as specified by state/project requirements.

4. Duplicate Samples

Performed at a minimum of every 20 samples, or as specified by state/project requirements.

5. Blank Analysis

a. Method Blank

If the concentration of the blank exceeds the MDL, all samples associated with the blank are redigested and reanalyzed concurrent with a new blank.

C. GAS CHROMATOGRAPHY/MASS SPECTROMETRY PROCEDURES

1. Calibration and Continuing Calibration

A minimum of an internal three point calibration is performed when indicated by the continuing calibration. One check standard is analyzed at the beginning of each 12-hour shift to verify calibration. The acceptance limit for the check standard is 25% RSD. Recalibration is necessary from once per week to once per month. Fresh calibration standards must be prepared weekly or as needed.

2. Validation of Mass Spectrometer

The mass spectrometers are tuned at the start of each run period and at 12-hour intervals. The tuning procedure utilizes the EPA/SW-846 recommended compounds 4-bromofluorobenzene (BFB) for 624/8240, 8260 and decafluorotriphenyl phosphine (DFTPP) for 625/8270.

3. Internal Standards

All sample results are quantified using the internal standard technique described in EPA methods 624, 8240, 8260, 625, 8270. Three (VOA) or six (BNA) internal standard compounds are added to each sample immediately before analysis. The internal standard nearest the retention time of the analyte of interest is used in the quantitation of the analyte.

4. Laboratory Control Sample

An EPA check sample (or external standard if EPA is unavailable) is analyzed at a minimum once every month. A standard is run every 12 hour shift.

5. Matrix Spike and Matrix Spike Duplicate

Performed at a minimum of every 20 samples or as specified by state/project requirements. This is the same procedure as the GC section.

6. Surrogate Spikes

Surrogate spiking compounds are added to and analyzed for, with every sample including blanks. A surrrogate is a compound chemically similar to a targeted analyte which is added to samples prior to purging or extraction.

B. GAS CHROMATOGRAPHY PROCEDURES

1. Calibration and Calibration Verification

All GC methods are calibrated by external calibration procedures using three to five standard concentrations, depending upon the method. A new calibration is performed at least once per quarter or as needed on routine analyses. Methods not utilized on a daily basis are calibrated before each run.

2. Laboratory Control Sample (LCS)

A second source external check sample is performed at a minimum of every 20 samples or as required by either state or project specific requirement or when a new calibration is performed.

3. Matrix Spike

Performed at a minimum of every 20 samples or as required by either state or project-specific requirements.

4. Surrogate Spike

Surrogates are added to and analyzed for in every sample per applicable organic methodologies.

5. Duplicate Sample Analysis

Performed at a minimum of every 20 samples or as specified by state/project requirements. The matrix spike is duplicated.

6. Blank Analysis

The reagent/method blank must have no contaminants greater than the detection limit of the method. In the case of volatile organic analysis, common laboratory solvents may be present at a concentration of less than 5 times the MDL.

7. Surrogate Spikes - Surrogate spiking compounds are added to and analyzed for, with every sample including blanks. A surrogate is a compound chemically similar to a targeted analyte which is added to samples prior to purging or extraction.

8. Other

Method 608/8080 are also subject to the following QC criteria:

a. Combined breakdown of endrin and DDT may not exceed 20%. This is monitored through the daily analysis of an LCS containing these compounds.

b. A CCV standard is extracted and analyzed after every tenth sample.

List of radiochemistry Methods (Cont.)

PACE	Americium, Isotopic
PACE	Plutonium, Isotopic
PACE	Thorium, Isotopic
PACE	Uranium, Isotopic
624/8240	Purgeable Volatiles ((GC/MS)
625/8270	BNA Extractable Semivolatiles (GC/MS)
608/8080	Organochlorine Pesticides and PCBs (GC/ECD)
615/8150	Phenoxy-Acid Herbicides (GC/ECD)
40 CFR 261	Characteristic of Ignitability
40 CFR 261	Characteristic of Corrosivity
40 CFR 261	Characteristic of Reactivity
40 CFR 261	TCLP
NIOSH 7400-A	Airborne Fibers (PCM)
600/M4-82-020	Bulk Asbestos (PLM)
NIOSH 0600	Nuisance Dust, Respirable
NIOSH 0500	Nuisance Dust, Total
NIOSH 7500	Respirable Silica (XRD)

4. List of Sample Preparation Methods

1311	TCLP
1312	Synthetic precipitation leaching procedure
3015	Microwave dig. aqueous
3051	Microwave dig. sludges, oil soil
3510	Separatory Funnel Liquid - Extraction
3520	Continuous Liquid - Extraction
3540	Soxhlet Extraction
3541	Automatic soxhlet extraction
3550	Sonication Extraction
3640	Gel Permeation Chromatography
3580	Waste Dilution
3630	Silica gel
3660	Sulfur clean up
5050	Bomb combus. method for T. Hal
5080	Purge and Trap
3005	Acid Digestion of Waters for Total Recoverable or Dissolved Metals for Analysis by Flame AA or ICP
3010	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by Flame AA or ICP
3020	Acid Digestion of Aqueous Samples and Extracts for Total Metals by Furnace AA
3050	Acid Digestion of Sediments, Soils, and Sludges

Method numbers refer to EPA Methods except:

5. Screening Methods

3810	Headspace
3820	Hexadecane extraction and screening of purgeable organics

1. S.M. = Standard Methods for the Examination of Water and Wastewater
2. USATHAMA = U.S. Army Toxic and Hazardous Materials Agency
3. NIOSH = Manual of Analytical Methods
4. Hach, Chevron, Calgon = Industrial Methods

6. List of Radiochemistry Methods

900.0	Gross Alpha/Beta
903.1	Radium-226
904.1	Radium-228
708	Tritium
901.1	Gamma Scan
704	Strontium-89/90
908.0	Total Uranium

Parameter	Method	NIOSH	OSHA
Yttrium	AA, ICP	7300	ID-121
Zinc	AA, ICP	7030, 7300	ID-121
C. Inorganic			
Acids, inorganic	IC	7903	
Ammonia	colorimetric	205	
	IC	6701	
Asbestos, bulk	PLM	9002	
Asbestos, fiber	TEM	7402	
Carbon black	gravimetric	5000	
Chlorine	colorimetric	209	ID-101
Cyanides, aerosol and gas	ISE	7904	
Fibers (including asbestos)	PCM	7400	
Fluorides, aerosol and gas	ISE	7902	
Hydrogen Peroxide	UV-VIS		V1-6
Hydrogen sulfide	UV-VIS	S-4	
Iodine	IC	6005	
Nitrogen dioxide	UV-VIS	6700,231	
Nuisance dust, respirable	gravimetric	0600	
Nuisance dust, total	gravimetric	0500	
Ozone	UV-VIS	S-8	
Silica	XRD	7500	
Stibine	colorimetric	6008	
Sulfuric acid	titration	S-174	
Sulfur dioxide	titration	163	
Wood dust	gravimetric	0500	

Parameter	Method	NIOSH	OSHA
Nicotine	GC	S-293	
Pentachlorophenol	HPLC-UV	5512	39
Phenol	HPLC		32
Phenyl Glycidyl Ether	GC-FID	S-74	
Polynuclear Aromatic	HPLC-UV	5506	
Polynuclear Hydrocarbons	GC-FID	5515	
Propylene glycol monomethyl ether	GC-FID		53
1,1,1,2-Tetrachloro-2,2-difluoroethane	GC-FID	1016	
1,1,2,2-Tetrachloro-1,2-difluoroethane	GC-FID	1016	
1,1,2,2-tetrachloroethane	GC-FID	1019	
Tetrahydrofuran	GC-FID	1609	
Trichloroethylene (TCE)	GC-FID	1022	
1,1,2-trichloro-1,2,2-trifluoroethane	GC-FID	1020	
Vinyl acetate	GC-FID	278	51
B. Metals			
Aluminum	AA, ICP	7013,7300	
Arsenic	AA, ICP		ID-105, 7300
Arsenic trioxide	GFAA	7901	
Arsine	GFAA	6001	
Barium	AA	7056	ID-121
Beryllium	GFAA, ICP	7102,7300	
Cadmium	AA, ICP	7048,7300	
Calcium	AA, ICP	7020,7300	ID-121
Chromium	AA, ICP	7024,7300	ID-121
Chromium, Hexavalent	UV-VIS	7600,S-317	
Cobalt	AA, ICP	7027,7300	
Copper	AA, ICP	7029,7300	
Indium	AA, ICP	173,7300	ID-121
Iron oxide	AA, ICP	173,7300	
Lead	AA, ICP	7082,7300	
Lithium	AA, ICP	173,7300	
Magnesium	AA, ICP	173,7300	
Manganese	AA, ICP	173,7300	ID-121
Molybdemium	AA, ICP	173,7300	ID-121
Nickel	AA, ICP	173,7300	ID-121
Palladium	AA	173	ID-121
Potassium	AA	173	ID-121
Selenium	AA		ID-105
Silver	AA, ICP	173,7300	ID-121
Sodium	AA, ICP	173,7300	
Strontium	AA	173	
Tellurium	AA, ICP	173,7300	ID-121
Thallium	AA, ICP	173,7300	
Tin - inorganic	AA, ICP	S-183,7300	ID-121
Titanium	AA, ICP	7300	ID-121
Tungsten	AA, ICP	7074,7300	
Vanadium	AA, ICP	173,7300	

4. Industrial Hygiene

A. Organic

Parameter	Method	NIOSH	OSHA
Acetic acid	GC-FID	1603	
Acrolein	GC-NPD		52
Acrylonitrile	GC-FID	1604	
Alcohols I	GC-FID	1400	
Alcohols II	GC-FID	1401	
Alcohols III	GC-FID	1402	
Alcohols IV	GC-FID	1403	
Amines,Aliphatic	GC-FID	221	
Aminoethanol	GC-FID	2007	
1,3-Butadiene	GC-FID	1024	
2-butanone (MEK)	GC-FID	2500	
n-butyl glycidyl ether	GC-FID	S-81	7
Dibutyl phthalate	GC-FID	5020	
Dichlorodifluoromethane	GC-FID	1018	
Dimethylacetmide	GC-FID	2004	
Dimethylforamide	GC-FID	2004	
Dioxane	GC-FID	1602	
Dipropylene glycol methyl ether	GC-FID	S-69	7
Endrin	GC-ECD	5519	
Epichlorohydrin	GC-FID	1010	
Estets I	GC-FID	1450	
Ethyl acetate	GC-FID	S-49	7
Ethylene dibromide	GC-FID	1008	
Ethylene glycol	GC-FID	5500	
Ethylene oxide	GC-ECD	1614	50
Ethyl ether	GC-FID	1610	
Fluorotrichloromethane	GC-FID	S-102	
Formaldehyde	GC-NPD		52
Glutaraldehyde	GC-FID	2531	
Glutaraldehyde	HPLC		64
Hydrazine	GC-FID/NPD	248	
Hydrocarbons	GC-FID	1500	7
Hydrocarbons,Aromatic	GGC-FID	1501	7
Hydrocarbons,Halogenated	GC-FID	1003	7
Isocyanates	HPLC-UV		42:47
Isophorone	GC-FID	2508	
Ketones I	GC-FID	1300	
Ketones II	GC-FID	1301	
Methanol	GC-FID	2000	
Methyl Cellosolve Acetate	GC-FID	S-39	
Methylene Chloride	GC-FID	1005	
Methyl Methacrylate	GC-FID	2537	
Mineral Oil mist	IR	5026	
Morpholine	GC	S-150	
Naphthas	GC-FID	1550	

Parameter	Method	Standard Methods 15th Ed.	ASTM
Viscosity	Saybolt		D88-81
% Water	Distillation		D95-83

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1982	SW 846
Sulfide, Total	Titration			9030
Reactive	Titration		261.23	Chap. 7 7.3.4.2
pH	Electrode			9040
Specific Conductance	Meter			9050 9045
Specific Gravity	Mass Displacement	213E		
Cyanide, Total	Pyridine-Barbitric Acid Colorimetic			9010
Amenable	Chlorination-Colorimetric			9010
Cyanide, Reactive	Pyridine-Barbitric Acid Colorimetric		261.23	Chap. 7 7.3.3.2
TCLP			40CFR268	1311

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1983	SW 846
Titanium	AA-Direct Aspiration	303C	283.1	
	AA-Furnace	304	283.2	
Vanadium	AA-Direct Aspiration	303C	286.1	7910
	AA-Furnace	304	286.2	7911
	ICP AES		200.7	6010
Zinc	AA-Direct Aspiration	303A	289.1	7950
	AA-Furnace	304	289.2	7951
	ICP AES		200.7	6010

3. **Wastes & Oil Analysis**

Parameter	Method	Standard Methods 15th Ed.	ASTM	SW 846
% Ash	Gravimetric	209F		
% Chlorine	Bomb Calorimeter		D808-81	
Density	Gravimetric	213E		
Flash Point Closed Cup	Tag		D93-80	1010
Free Liquids	Paint Filter			9095
Heat of Combustion	Bomb Calorimeter		D240-76	
Leach Test. EP Toxicity	Extraction			1310
ASTM Water	Extraction		D3987-85	
% Sulfur	Bomb Calorimeter		D129-64	

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1983	SW 846
Lithium	AA-Direct Aspiration	317B		
Magnesium	AA-Direct Aspiration	303A	242.1	7450
	ICP AES		200.7	6010
Manganese	AA-Direct Aspiration	303A	243.1	7460
	AA-Furnace	304	243.2	7461
	ICP AES		200.7	6010
Mercury	AA-Cold Vapor	303F	245.1	7470 or 7471
Molybdenum	AA-Direct Aspiration	303C	246.1	7480
	AA-Furnace	304	246.2	7481
Nickel	AA-Direct Aspiration	303A	249.1	7520
	AA-Furnace	304	249.2	
	ICP AES		200.7	6010
Potassium	AA-Direct Aspiration	303A	258.1	7610
Selenium	AA-Gaseous Hydride	303E	270.3	7740
	AA-Furnace	304	270.2	7741
	ICP AES		200.7	6010
Silver	AA-Direct Aspiration	303A	272.1	7760
	AA-Furnace	304	272.2	7761
	ICP AES		200.7	6010
Sodium	AA-Direct Aspiration	303A	273.1	7770
	ICP AES		200.7	6010
Strontium	AA-Direct Aspiration	303A		7780
Thallium	AA-Direct Aspiration	303A	279.1	7840
	AA-Furnace	304	279.2	7841
	ICP AES		200.7	6010
Tin	AA-Direct Aspiration	303A	282.1	7870
	AA-Furnace	304	282.2	

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1983	SW 846
Barium	AA-Direct Aspiration	303C	208.1	7080
	AA-Furnace	304	208.2	7081
	ICP-AES		200.7	6010
Beryllium	AA-Direct Aspiration	303C	210.1	7090
	AA-Furnace	304	210.2	7091
	ICP-AES		200.7	6010
Cadmium	AA-Direct Aspiration	303A	213.1	7130
	AA-Furnace	304	213.2	7131
	ICP-AES		200.7	6010
Calcium	AA-Direct Aspiration	303A	215.1	7140
	AA-Furnace	311C	215.2	
	ICP-AES		200.7	6010
Chromium,				
Total	AA-Direct Aspiration	303A	218.1	7190
Hexavalent	AA-Furance	304	218.2	7191
	ICP AES		200.7	6010
	Colorimetric	312B		7196
	MIBK Extraction			7197
Cobalt	AA-Direct Aspiration	303A	219.1	7200
	AA-Furnace	304	219.2	7201
	ICP-AES		200.7	6010
Copper	AA-Direct Aspiration	303A	220.1	7210
	AA-Furnace	304	220.2	7211
	ICP-AES		200.7	6010
Iron	AA-Direct Aspiration	303B	236.1	7380
	AA-Furnace	304	236.2	7381
	ICP-AES		200.7	6010
Lead	AA-Direct Aspiration	303A	239.1	7420
	AA-Furnace	304	239.2	7421
	ICP-AES		200.7	6010

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1983	ASTM	SW 846
Specific Conductance	Meter	205	120.1	D1125	9040
Sulfate	Ion Chromatography	426C	300.0	D516	9035
	Automated Methyl Thymol Blue		375.2		9038 9036
	Turbidimetric		375.4		
Sulfide	Colorimetric	427C	376.2		9030
	Titration	427D	376.1		
Sulfite	Titration	428A	377.1	D1339	
Surfactants (MBAS)	Methylene Blue	512B	425.1	D2330	
Turbidity	Meter	214A	180.1	D1889	

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1979	SW 846
B. Metals				
Aluminum	AA-Direct Aspiration	303C	202.1	7020
	AA-Furnace	304	202.2	
	ICP-AES		200.7	6010
Antimony	AA-Direct Aspiration	303A	204.1	7040
	AA-Furnace	304	204.2	7041
	ICP-AES		200.7	6010
Arsenic	AA-Gaseous Hydride	303E	206.3	7061
	AA-Furnace	304	206.2	7060
	ICP-AES		200.7	6010

Parameter	Method	Standard Methods 15th Ed.	EPA Methods	ASTM	SW 846
Hardness, Total	EDTA Titration Calculation	314B	130.2	D1126	
Hardness, Calcium	EDTA Titration	303A	242.1	D511	
Ion Chromatography			300.0		9056
Nitrogen,					
Ammonia	Distillation Titration	417D	350.2		
	Potentiometric		350.3		
Kjeldahl	Digestion Distillation	420B	351.3	D3590	
Nitrate	Automated Cadmium	418F	353.2	D3867	9200
	Brucine Sulfate		352.1	D091	
Nitrite	Automated Cadmium	418F	353.2	D3867	
	Colorimetric	419			
Organic	Kjeldahl-NH_3	420A	351.3	D3590	
	Kjeldahl-Potentiometric		351.4		
Oil & Grease	Soxhlet	503C			
	Partition-Gravimetric	503A	413.1		9070/
			413.2		9071
Oxygen	Winkler	421B	360.2	D888	
Dissolved	Electrode	421F	360.1		
pH (Hydrogen Ion)	Electrode	423	150.1	D1293	9040 9045
Phenol	Distillation-Extraction Colorimetric		420.1	D1783	9066 9065 9069
Phosphorus,	Persulfate Digestion-	424C/F	365.2	D515	
Total	Ascorbic Acid Reduc.				
Ortho	Ascorbic Acid Reduc.	424F	365.2	D515	
Silica,	Molybdosilicate	425C	370.1	D859	
Dissolved	ICP		200.7		
Solids					
Total	Gravimetric	209A	160.3		
Total Volatile	Gravimetric	209D	160.4		
Suspended	Gravimetric	209C	160.2		
Suspended Volatile	Gravimetric	209D	160.4		
Total Dissolved	Gravimetric	209B	160.1		
Settleable	Gravimetric	209E	160.5		
TOC			415.1		9060
TOX					9022

2. Inorganic Analyses

Parameter	Method	Standard Methods 15th Ed.	EPA Methods 1983	ASTM	SW 846
A. Non Metals					
Acidity	Potentiometric Titration	402	305.1	D1067	
Alkalinity	Potentiometric Titration	403	310.1	D1067	
Bacteria, Total Coliform	Membrane Filter	909A			9132/9131
Fecal Coliform	Membrane Filter	908C			
Fecal Strept.	Membrane Filter	910A			
Total Plate Count	Agar Medium	907			
Biochemical Oxygen Demand, 5-Day	Winkler Electrode	507 507	405.1		
Boron	Curcumin 405-A	404A	212.3		
	ICP		200.7		6010
Chemical Oxygen Demand	Dichromate Reflux (High)	508A	410.1	D1252	
	Dichromate Reflux (Low)	508A	410.2	D1252	
Chloride	Mercuric Nitrate	407B	325.3	D512	9252
	Auto. Ferricyanide	407D	325.2		9251
	Titration	407A			
Chlorine, Residual	Amperometric Titration	408C	330.1 330.5	D1253	
	Colorimetric	408E			
Color	Visual Comparison	204A	110.2		
Cyanide, Total	Pyridine-Barbitutic Acid, Colorimetric	412D	335.2	D2036	9010A
Amenable	Chlorination-Colorimetric	412F	335.1	D2036	9010A 9012
Flouride, Total	Distillation-Electrode	413A/B	340.2	D1179	
Flouride, Diss.	Electrode	413B	340.2	D1179	

Parameter	Method	DW	WW Method	SW 846	Spec.
Chlorinated Hydrocarbons	GC		612	8120	
2, 3, 7, 8 - TCDD	GC/MS		613		
Volatile Organics					(MN)465C
Base/Neutrals & Acids	GC/MS	525 (NCA)	625	8250/8270	
Organophosphorus Pesticides	GC	507	614/622	8140/ 8220	(MN)570A
Chlorinated Herbicides	GC	515.1	615/608.1/ 608.2	8150	(MN)574A (CA)509B
EDB and DBCP	GC	504			(CA) DOHS/ 8011
Volatile Organic Compounds	GC/MS	524.2/524.1	624	8240	B260
Carbamates & Urea & Pesticides	HPLC	531.1	632		(MN)572A (MN)572A
Fuel Hydrocarbons & BTEX	GC or IR		602/418.1	8020	(CA)9073 Mod. 8015
Alachlor, Atrazine	GC	507/505	619/645		(MN)570A (MN)570A
Chlordane, Heptachlor, Heptachlor Expoxide, Lindane; Methoxychlor	GC	508/505	608/617	8080	8081
Aldicarb; Aldicarb sulfone; Aldicarb sulfoxide; Carbofuran	GC	531.1			

IX. ANALYTICAL PROCEDURES

Analytical methods used at PACE are EPA methodologies, where available, such as those specified in References 2 and 3 or approved equivalent methods. A list of typical analytical methods utilized at PACE is as follows:

A. LIST OF ANALYTICAL METHODS

1. Organic Analyses

Parameter	Method	DW	WW Method	SW 846	Spec.
Purgeable Halocarbons	GC	502.1/502.2	601	8010	
Non-Halogenated Volatile Organics	GC			8015	
Purgeable Aromatics and Unsaturated Organics	GC	503.1/502.2	602	8020	
Acrolein & Acrylonitrile	GC		603	8030	
Phenols	GC	515.1	604	8040	
Benzidines	HPLC		605		
Phthalate Esters	GC		606	8060	
Nitrosamines	GC		607		
Organochlorine Pesticides and PCBs	GC	508/505/508A 507/515.1	608/608.1 608.2	8080	(CA)Mod 8080 (MN)570A
Nitroaromatics and Isophorone	GC		609	8090	
Polynuclear Aromatic Hydrocarbons	HPLC/GC		610	8310/ 8100	
Haloethers	GC		611		
Alachlor, Atrazine, Chlordane, Hepatchlor, Heptachlor Epoxide, Lindane, Methoxychlor, Toxaphene, and PCBs (as Aroclors)	GC	505/507	645		MN 570A

EXHIBIT 13

CONTINUING CALIBRATION CHECK
Semi-Volatile Compounds

CASE NO:_____ CALIBRATION DATE:_____

LABORATORY NAME: PACE LABORATORIES TIME:_____

CONTRACT/PROJECT NO._____ ANALYST:_____

INSTRUMENT I.D.:_____ INITIAL CALIBRATION DATE:___

MAXIMUM %D FOR CCC IS 10%

COMPOUND	CF	CF	%D	CCC
Alpha-BHC				
* Beta-BHC				
Lindane				
Delta-BHC				
Heptachlor				
* Aldrin				
Heptachlor Epoxide				
* Endosulfan I				
DDF/Dieldrin				
* Endrinion				
Endosulfan II				
4,4'-DDD				
Endrin Aldehyde				
4,4'-DDT				
Endosulfan Sulfate				
Aroclor 1016				
Aroclor 1221				
Aroclor 1232				
Aroclor 1242				
Aroclor 1248				
Aroclor 1254				
Aroclor 1260e				
Chlordane				
Toxaphene				
Methoxychor				
DBC				

CF -Calibration Factor from daily standard at ug/L
CF-Average Calibration Factor from initial calibration Form VI
%D-Percent Difference
CCC-Calibration Check Compounds

INITIAL CALIBRATION DATA
EXTRACTABLE 8080/608 COMPOUNDS
EXHIBIT 12

CALIBRATION DATE: COLUMN ID:

INSTRUMENT ID: DETECTOR ID:

MAXIMUM % RSD IS 20%

Standard ID	/	/	/		
Compound	CF 20	CF 40	CF 60	$\overline{CF}$	%RSD
Alpha-BHC					
Beta- BHC					
Lindane					
Delta- BHC					
Heptachlor					
Aldrin					
Heptachlor Epoxide					
Endosulfan I					
DDE/Dieldrin					
Endrin					
Endosulfan II					
4,4'-DDD					
Endrin Aldehyde					
4,4'-DDT					
Endosulfan Sylfate					

CF=CALIBRATION FACTOR= $\frac{\text{Total ng of Standard}}{\text{Area}}$

$\overline{CF}$ = AVERAGE CALIBRATION FACTOR = CF/n

%RSD = RELATIVE STANDARD DEVIATION = $\frac{\text{(Standard Dev.) (100)}}{\overline{CF}}$

TABLE 2. QC ACCEPTANCE CRITERIA[a] FOR GC/MS SEMIVOLATILE ORGANICS (CONT.)

Parameter	Test conc. (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range p, p_s (%)
Indeno(1,2,3-cd)pyrene	100	44.6	D-150.9	D-171
Isophorone	100	63.3	46.6-180.2	21-196
Naphthalene	100	30.1	35.6-119.6	21-133
Nitrobenzene	100	39.3	54.3-157.6	35-180
N-Nitrosodi-n-propylamine	100	55.4	13.6-197.9	D-230
PCB-1260	100	54.2	19.3-121.0	D-164
Phenanthrene	100	20.6	65.2-108.7	54-120
Pyrene	100	25.2	69.6-100.0	52-115
1,2,4-Trichlorobenzene	100	28.1	57.3-129.2	44-142
4-Chloro-3-methylphenol	100	37.2	40.8-127.9	22-147
2-Chlorophenol	100	28.7	36.2-120.4	23-134
2,4-Chlorophenol	100	26.4	52.5-121.7	39-135
2,4-Dimethylphenol	100	26.1	41.8-109.0	32-119
2,4-Dinitrophenol	100	49.8	D-172.9	D-191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0-100.0	D-181
2-Nitrophenol	100	35.2	45.0-166.7	29-182
4-Nitrophenol	100	47.2	13.0-106.5	D-132
Pentachlorophenol	100	48.9	38.1-151.8	14-176
Phenol	100	22.6	16.6-100.0	5-112
2,4,6-Trichlorophenol	100	31.7	52.4-129.2	37-144

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

p, p_s = Percent recovery measured.

D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 625. These criteria are based directly on the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7.

TABLE 2. QC ACCEPTANCE CRITERIA[a] FOR GC/MS SEMIVOLATILE ORGANICS

Parameter	Test conc. (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_s (%)
Acenaphthene	100	27.6	60.1-132.3	47-145
Acenaphthylene	100	40.2	53.5-126.0	33-145
Aldrin	100	39.0	7.2-152.2	D-166
Anthracene	100	32.0	43.4-118.0	27.133
Benzo(a)anthracene	100	27.6	41.8-133.0	33-143
Benzo(b)fluoranthene	100	38.8	42.0-140.4	24-159
Benzo(k)fluoranthene	100	32.3	25.2-145.7	11-162
Benzo(a)pyrene	100	39.0	31.7-148.0	17-163
Benzo(ghi)perylene	100	58.9	D-195.0	D-219
Benzyl butyl phthalate	100	23.4	D-139.9	D-152
β-BHC	100	31.5	41.5-130.6	24-149
δ-BHC	100	21.6	D-100.0	D-110
Bis(2-chloroethyl)ether	100	55.0	42.9-126.0	12-158
Bis(2-chloroethoxy)methane	100	34.5	49.2-164.7	33-184
Bis(2-chloroisopropyl)ether	100	46.3	62.8-138.6	36-166
Bis(2-ethylhexyl)phthalate	100	41.1	28.9-136.8	8-158
4-Bromophenyl phenyl ether	100	23.0	64.9-114.4	53-127
2-Chloronaphthalene	100	13.0	64.5-113.5	60-118
4-Chlorophenyl phenyl ether	100	33.4	38.4-144.7	25-158
Chrysene	100	48.3	44.1-139.9	17-168
4,4'-DDD	100	31.0	D-134.5	D-145
4,4'-DDE	100	32.0	19.2-119.7	4-136
4,4'-DDT	100	61.6	D-170.6	D-203
Dibenzo(a,h)anthracene	100	70.0	D-199.7	D-227
Di-n-butyl phthalate	100	16.7	8.4-111.0	1-118
1,2-Dichlorobenzene	100	30.9	48.6-112.0	32-129
1,3-Dichlorobenzene	100	41.7	16.7-153.9	D-172
1,4-Dichlorobenzene	100	32.1	37.3-105.7	20-124
3,3'-Dichlorobenzidine	100	71.4	8.2-212.5	D-262
Dieldrin	100	30.7	44.3-119.3	29-136
Diethyl phthalate	100	26.5	D-100.0	D-114
Dimethyl phthalate	100	23.2	D-100.0	D-112
2,4-Dinitrotoluene	100	21.8	47.5-126.9	39-139
2,6-Dinitrotoluene	100	29.6	68.1-136.7	50-158
Di-n-octylphthalate	100	31.4	18.6-131.8	4-146
Endosulfan sulfate	100	16.7	D-103.5	D-107
Endrin aldehyde	100	32.5	D-188.8	D-209
Fluoranthene	100	32.8	42.9-121.3	26-137
Fluorene	100	20.7	71.6-108.4	59-121
Heptachlor	100	37.2	D-172.2	D-192
Heptachlor epoxide	100	54.7	70.9-109.4	26.155
Hexachlorobenzene	100	24.9	7.8-141.5	D-152
Hexachlorobutadiene	100	26.3	37.8-102.2	24-116
Hexachloroethane	100	24.5	55.2-100.0	40-113

TABLE 2. CALIBRATION AND QC ACCEPTANCE CRITERIA[a] FOR GC/MS VOLATILE ORGANI

Parameter	Range for Q (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_s (%)
Benzene	12.8-27.2	6.9	15.2-26.0	37-151
Bromodichloromethane	13.1-26.9	6.4	10.1-28.0	35-155
Bromoform	14.2-25.8	5.4	11.4-31.1	45-169
Bromomethane	2.8-37.2	17.9	D-41.2	D-242
Carbon tetrachloride	14.6-25.4	5.2	17.2-23.5	70-140
Chlorobenzene	13.2-26.8	6.3	16.4-27.4	37-160
2-Chloroethylvinyl ether	D-44.8	25.9	D-50.4	D-305
Chloroform	13.5-26.5	6.1	13.7-24.2	51-138
Chloromethane	D-40.8	19.8	D-45.9	D-273
Dibromochloromethane	13.5-26.5	6.1	13.8-26.6	53-149
1,2-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,3-Dichlorobenzene	14.6-25.4	5.5	17.0-28.8	59-156
1,4-Dichlorobenzene	12.6-27.4	7.1	11.8-34.7	18-190
1,1-Dichloroethane	14.5-25.5	5.1	14.2-28.4	59-155
1,2-Dichloroethane	13.6-26.4	6.0	14.3-27.4	49-155
1,1-Dichloroethene	10.1-29.9	9.1	3.7-42.3	D-234
trans-1,2-Dichloroethene	13.9-26.1	5.7	13.6-28.4	54-156
1,2-Dichloropropane	6.8-33.2	13.8	3.8-36.2	D-210
cis-1,3-Dichloropropene	4.8-35.2	15.8	1.0-39.0	D-22
trans-1,3-Dichloropropene	10.0-30.0	10.4	7.6-32.4	17-18
Ethyl benzene	11.8-28.2	7.5	17.4-26.7	37-162
Methylene chloride	12.1-27.9	7.4	D-41.0	D-221
1,1,2,2-Tetrachloroethane	12.1-27.9	7.4	13.5-27.2	46-157
Tetrachloroethene	14.7-25.3	5.0	17.0-26.6	64-148
Toluene	14.9-25.1	4.8	16.6-26.7	47-150
1,1,1-Trichloroethane	15.0-25.0	4.6	13.7-30.1	52-162
1,1,2-Trichloroethane	14.2-25.8	5.5	14.3-27.1	52-150
Trichloroethene	13.3-26.7	6.6	18.5-27.6	71-157
Trichlorofluoromethane	9.6-30.4	10.0	8.9-31.5	17-181
Vinyl chloride	0.8-39.2	20.0	D-43.5	D-251

Q = Concentration measured in QC check sample, in ug/L.
s = Standard deviation of four recovery measurements, in ug/L.
$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.
P, P_s = Percent recovery measured.
D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 624 and were calculated assuming a QC check sample concentration of 20 ug/L. These criteria are based directly upon the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 7.

TABLE 2. QC ACCEPTANCE CRITERIA[a] FOR ORGANOCHLORINE PESTICIDES & PCB's

Parameter	Test conc. (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_s (%)
Aldrin	2.0	0.42	1.08-2.24	42-122
α-BHC	2.0	0.48	.98-2.44	37-134
β-BHC	2.0	0.64	0.78-2.60	17-147
δ-BHC	2.0	0.72	1.01-2.37	19-140
γ-BHC	2.0	0.46	0.86-2.32	32-127
Chlordane	50	10.0	27.6-54.3	45-119
4,4'-DDD	10	2.8	4.8-12.6	31-141
4,4'-DDE	2.0	0.55	1.08-2.60	30-145
4,4'-DDT	10	3.6	4.6-13.7	25-160
Dieldrin	2.0	0.76	1.15-2.49	36-146
Endosulfan I	2.0	0.49	1.14-2.82	45-153
Endosulfan II	10	6.1	2.2-17.1	D-202
Endosulfan Sulfate	10	2.7	3.8-13.2	26-144
Endrin	10	3.7	5.1-12.6	30-147
Heptachlor	2.0	0.40	0.86-2.00	34-111
Heptachlor epoxide	2.0	0.41	1.13-2.63	37-142
Toxaphene	50	12.7	27.8-55.6	41-126
PCB-1016	50	10.0	30.5-51.5	50-114
PCB-1221	50	24.4	22.1-75.2	15-178
PCB-1232	50	17.9	14.0-98.5	10-215
PCB-1242	50	12.2	24.8-69.6	39-150
PCB-1248	50	15.9	29.0-70.2	38-158
PCB-1254	50	13.8	22.2-57.9	29-131
PCB-1260	50	10.4	18.7-54.9	8-127

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

P, P_s = Percent recovery measured.

D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 608. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

TABLE 2. CALIBRATION AND QC ACCEPTANCE CRITERIA[a] FOR AROMATIC VOLATILE ORGANIC

Parameter	Range for Q (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_s (%)
Benzene	15.4-24.6	4.1	10.0-27.9	39-150
Chlorobenzene	16.1-23.9	3.5	12.7-25.4	55-135
1,2-Dichlorobenzene	13.6-26.4	5.8	10.6-27.6	37-154
1,3-Dichlorobenzene	14.5-25.5	5.0	12.8-25.5	50-141
1,4-Dichlorobenzene	13.9-26.1	5.5	11.6-25.5	42-143
Ethylbenzene	12.6-27.4	6.7	10.0-28.2	32-160
Toluene	15.5-24.5	4.0	11.2-27.7	46-148

Q = Concentration measured in QC check sample, in ug/L.

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

P, P_s = Percent recovery measured.

[a]Criteria are from 40 CFR Part 136 for Method 602 and were calculated assuming a QC check sample concentration of 20 ug/L. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 1.

TABLE 2. CALIBRATION AND QC ACCEPTANCE CRITERIA[a] FOR AROMATIC VOLATILE ORGANICS

Parameter	Range for Q (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_s (%)
Benzene	15.4-24.6	4.1	10.0-27.9	39-150
Chlorobenzene	16.1-23.9	3.5	12.7-25.4	55-135
1,2-Dichlorobenzene	13.6-26.4	5.8	10.6-27.6	37-154
1,3-Dichlorobenzene	14.5-25.5	5.0	12.8-25.5	50-141
1,4-Dichlorobenzene	13.9-26.1	5.5	11.6-25.5	42-143
Ethylbenzene	12.6-27.4	6.7	10.0-28.2	32-160
Toluene	15.5-24.5	4.0	11.2-27.7	46-148

Q = Concentration measured in QC check sample, in ug/L.

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

P, P_s = Percent recovery measured.

[a]Criteria are from 40 CFR Part 136 for Method 602 and were calculated assuming a QC check sample concentration of 20 ug/L. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 1.

TABLE 2 CALIBRATION AND QC ACCEPTANCE CRITERIA[a] FOR HALOGENATED VOLATILE ORGAN

Parameter	Range for Q (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_s (%)
Bromodichloromethane	15.2-24.8	4.3	10.7-32.0	42-172
Bromoform	14.7-25.3	4.7	5.0-29.3	13-159
Bromomethane	11.7-28.3	7.6	3.4-24.5	D-144
Carbon tetrachloride	13.7-26.3	5.6	11.8-25.3	43-143
Chlorobenzene	14.4-25.6	5.0	10.2-27.4	38-150
Chloroethane	15.4-24.6	4.4	11.3-25.2	46-137
2-Chloroethylvinyl ether	12.0-28.0	8.3	4.5-35.5	14-186
Chloroform	15.0-25.0	4.5	12.4-24.0	49-133
Chloromethane	11.9-28.1	7.4	D-34.9	D-193
Dibromochloromethane	13.1-26.9	6.3	7.9-35.1	24-191
1,2-Dichlorobenzene	14.0-26.0	5.5	1.7-38.9	D-208
1,3-Dichlorobenzene	9.9-30.1	9.1	6.2-32.6	7-187
1,4-Dichlorobenzene	13.9-26.1	5.5	11.5-25.5	42-143
1,1-Dichloroethane	16.8-23.2	3.2	11.2-24.6	47-132
1,2-Dichloroethane	14.3-25.7	5.2	13.0-26.5	51-147
1,1-Dichloroethene	12.6-27.4	6.6	10.2-27.3	28-167
trans-1,2-Dichloroethene	12.8-27.2	6.4	11.4-27.1	38-155
1,2-Dichloropropane	14.8-25.2	5.2	10.1-29.9	44-156
cis-1,3-Dichloropropene	12.8-27.2	7.3	6.2-33.8	22-178
trans-1,3-Dichloropropene	12.8-27.2	7.3	6.2-33.8	22-178
Methylene chloride	15.5-24.5	4.0	7.0-27.6	25-162
1,1,2,2-Tetrachloroethane	9.8-30.2	9.2	6.6-31.8	8-184
Tetrachloroethene	14.0-26.0	5.4	8.1-29.6	26-162
1,1,1-Trichloroethane	14.2-25.8	4.9	10.8-24.8	41-138
1,1,2-Trichloroethane	15.7-24.3	3.9	9.6-25.4	39-136
Trichloroethene	15.4-24.6	4.2	9.2-26.6	35-146
Trichlorofluoromethane	13.3-26.7	6.0	7.4-28.1	21-156
Vinyl chloride	13.7-26.3	5.7	8.2-29.9	28-163

Q = Concentration measured in QC check sample, in ug/L.

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

P, P_s = Percent recovery measured.

D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 601 and were calculated assuming a QC check sample concentration of 20 ug/L.

VIII. CALIBRATION PROCEDURES AND FREQUENCY

Most measurements taken in the laboratory are based upon comparison to reference standards as analyzed by the standard method. The standard results are utilized to generate calibration curves or calibration factors. The results of the sample analysis are then quantified.

All instruments are calibrated using standard solutions of known concentrations. The standards are prepared from certified reference materials and are generally traceable back to NIST. Refer to Section XI for additional information.

The laboratory calibration procedures utilized meet or exceed the method calibration criteria for all analyses performed. If the method calibration requirements are more stringent than discussed in this document, the more stringent calibration requirements shall be achieved. The minimum instrument calibration procedures are discussed in Section IX by major instrument group; the calibration procedure in the method is followed for each specific analysis. Calibration procedures are documented on computer generated printouts and benchsheets where applicable.

Continuous calibration is verified by analysis of calibration standards or laboratory control samples from different sources at regular intervals. Recalibration is performed at specified time intervals or when indicated by the continuous verification procedure or as required by the method. Typical calibration and QC acceptance criteria for some common organic analyses are summarized in Table 2.

Forms to document initial and continuing calibration have been developed (Exhibits 12 and 13).

Refer to Section IX for additional calibration information and frequency as specified in the specific analytical methods.

EXHIBIT 11

HAZARDOUS MATERIAL/WASTE SAMPLE DISPOSAL OPTION FORM

Client _____ Client Project ID _____

Contact _____ PACE Project # _____

Address _____ Project Manager _____

_____ Report Sent Date _____

Phone Number _____ Pull Sample Date _____

Sample #	Matrix	Location	Disposal Method	Charge

Remarks: 1 = Return to Client
2 = In House Disposal
C = Clean
D = Dirty

Removed from Refrigerator (initial/date) _____

Returned to Client (initial/date) _____

Disposed of Samples (initial/date _____

- The shipping clerk labels the box with an appropriate hazard label and ships the samples back to the client using UPS or any other requested manner for shipment. (Note: It is important for proper packaging to prevent breakage during shipment.)

- All shipping costs will be charged against the appropriate project number.

d. Upon receipt of Sample Disposition Form, the file clerk files it with other project related information.

4. Hazardous Material/Waste Sample Disposition Option

The preferred method for disposition of excess hazardous material/waste samples is to return the excess sample to the client. It may not be feasible to return samples in all cases or the client may require PACE to dispose of excess samples. PACE will dispose of excess hazardous samples when required and will charge a disposal fee to recover costs for management and disposal.

Procedure for Disposal Option for Excess Hazardous Material/Waste Samples:

a. The project manager informs the client that excess sample disposal will require an additional charge.

b. When analyses are complete, the project manager indicates disposal as the option on the Sample Disposition Form and completes and attaches Hazardous Material/Waste Disposal Option Form (Exhibit 11). An entry is to be made in all fields of this form as it will determine the basis for lab packing and disposal.

c. The project manager routes the Disposal Option Form to sample check-in.

d. The project manager is responsible for billing the client for disposal.

e. The sample custodian is responsible for maintaining a file of Disposal Option Forms for all samples awaiting disposal. Hazardous material/waste samples are stored in safe manner, segregated by compatibility groups as indicated by the hazardous waste disposal SOP.

f. The Quality Control Manager is responsible for reviewing accumulated samples awaiting disposal and initiating the disposal process when warranted. The Field Services, Inorganic, Organic, and Environmental Services Departments cooperate and participate in the disposal process. (For compatibility and compositing, see the Hazardous Waste Disposal SOP.)

All Extracted/Tainted Samples

CAM Extracts - Clean - Neutralize/sink
Dirty - Acid metals waste

Other Extracts - Toxic waste

Liquid/Unknown Misc. - Project manager specify

- Project manager will complete the sample disposition form and route it back to invoicing.
- The invoicing department will put completed sample disposition form in sample control mailbox.

b. Upon receipt of the Sample Disposition Form by the sample custodian personnel, the custodian personnel will remove the samples from storage using the information provided on the form.

- If the Sample Disposition Form indicates "Dump," the sample custodian personnel will remove them from storage and place them at a sample disposal station for proper disposal. The process of disposal is performed by the sample custodian personnel or appropriate laboratory staff. The Sample Disposition Form is signed and dated by the sample custodian personnel, then routed to the file clerk for filing with other project information.
- If the samples are to be returned, the sample custodian removes the sample or samples from storage, initials and dates the Sample Disposition Form. The samples, the Sample Disposition Form, and a copy of the client's chain-of-custody are then delivered to the shipping clerk by the sample custodian for return to the client.

c. Upon receipt of the samples and Sample Disposition Form, the shipping clerk signs and dates the form.

The Sample Disposition Form is copied and the original form with the samples is returned to the client, along with a copy of the client's chain-of-custody. A copy of the Sample Disposition Form and the original chain-of-custody is routed to the file clerk for filing with other project information (QC file).

3. Procedure for Use of the Sample Disposition Form

a. The project manager separates the sample disposition form from the report package, signs the form, and routes it to the sample custodian. If the sample is water or wastewater and non-hazardous, the project manager may wish to properly dispose of the waste.

- If the project requires, the project manager may hold the form for an acceptable amount of time before return or disposal.

- It is important that this form be used and not discarded. It is part of the internal chain-of-custody and is filed with the project report.

- The project manager will use action codes such as:

1 = Return to client
2 = In house disposal
C = Clean
D = Dirty

As a general rule, soil samples will be returned and water samples will be disposed of in-house. Water samples which are highly contaminated will be returned. Preserved samples, VOA's, and extracted/tainted samples will not be returned to the client. Therefore, it is necessary to note clean or dirty to facilitate handling. If a sample has an extremely high level of contamination, note the contaminant.

For In-House Sample Disposal

All preserved - Clean - Neutralize/sink
Dirty - Toxic waste

Un-preserved water - Clean - Sink
Dirty - Toxic waste

Soil/Sludge - Clean - Trash
Dirty - Toxic waste

All VOA's - Clean - Neutralize/sink
Dirty - Toxic waste

EXHIBIT 10

SAMPLE DISPOSITION FORM

_____

_____

_____

_____

_____

Date removed: _____
Initials: _____

Date shipped: _____
Initials: _____

RE: Client Project ID: _____

PACE Project No.: _____

Sample ID _____ _____ _____ _____

Dear _____:

All requested analyses of the samples for the above referenced project have been completed. Enclosed are the remaining portions of the samples which are being returned to you for final disposition.

If you have any questions, please call me.

Sincerely,

_____
Project Manager

pace,
INCORPORATED
THE ASSURANCE OF QUALITY

EXHIBIT 9

August 29, 1991

Dear Valued Client:

A new policy has been implemented in the Sample Receiving Department of PACE, Inc. We hope that this policy will be helpful to you.

Upon receipt of samples into the laboratory, the Sample Custodian completes a Sample and Analysis Data Entry Form. This form is designed to accommodate a short description of the samples received (sample name and/or sample reference), the type of container, and a list of the analyses requested to be performed on each sample. A copy of this form will be sent to the Client (submitter).

Enclosed is a copy of the Sample and Analysis Data Entry Form relevant to the samples we recently received from you. Please compare the information on the form to assure that it is consistent with your request. If there is any inconsistency or if you have any questions on your project, please call the PACE Contact indicated on the Sample and Analysis Data Entry Form. The PACE Contact has primary responsibility for monitoring the progress of your project through the laboratory.

It is also part of PACE, Inc.'s Standard Operating Procedure to return all samples pertaining to the information attached that are hazardous materials or hazardous wastes to the client at project completion. PACE, Inc. reserves the right to return or dispose of all samples at our discretion.

We have implemented this procedure to better serve our clients, and would appreciate any comments you may have.

Sincerely,

1710 Douglas Drive North
Minneapolis, MN 55422
TEL: 612 544-5543
FAX 612 525 3377

1. PACE, Inc. Standard Operating Procedure is to return all samples of hazardous materials or wastes to the client at project completion. PACE, Inc. reserves the right to return or dispose of all samples at our discretion. (Exhibit 9) This is a pre-printed cover letter that accompanies the Sample and Analysis Data Entry Form.

d. The Sample and Analysis Data Entry Form and cover letter is sent to the project manager and to the client by the sample custodian personnel.

2. <u>Sample Return and Disposal</u>

Upon completion of laboratory analysis and/or the project, the LDMS automatically prints a report, invoice and sample disposition form. This form is part of the report package and is routed to the project manager.

a. The Sample Disposition Form (Exhibit 10) contains the following information:

1. Client name, address, and contact
2. PACE project number
3. Client project identification number
4. PACE sample identification number
5. PACE project manager name

2. Responsibilities for SOP Compliance

a. The QC manager has the overall responsibility for ensuring that the SOP is implemented and followed.

b. The sample custodian personnel have the responsibility for ensuring that the SOP is properly followed, and to notify the QC manager of problems.

c. All employees checking out samples are required to follow procedures.

G. EXCESS SAMPLE DISPOSITION

Samples not totally consumed during the analyses are returned to the client. It is the project manager's responsibility to ensure that proper disposal has taken place. If the sample is water or wastewater and is considered non-hazardous by the project manager, it may then (by request) be properly disposed of at PACE facilities and not returned to the client.

1. Notification of Sample Return

The project manager and client receive written notification at the time of project initiation in the following manner:

a. The project proposal states the following paragraph in its Conditions and Terms Statement:

PACE, Inc. Standard Operating Procedures is to return all samples of hazardous materials or wastes to the client at project completion, and PACE, Inc. reserves the right to return or dispose of all samples at our discretion.

This is a standard form used by PACE's Marketing Department.

b. The Sample and Analysis Data Entry Form states the following sentence:

- PACE, Inc. reserves the right to return all samples at our discretion.

- This form is printed out by the LDMS at sample check-in.

c. The Sample and Analysis Data Entry Form cover letter will state the following paragraph:

EXHIBIT 8

SAMPLE CUSTODY LOG

Contract/Project No.: _____
Date Received: _____
Received by: _____
Time: _____
Witness: _____
Stored in: _____

Samples No(s). _____
_____
_____
_____
_____
_____

Date & Time Removed	Sample Nos. Removed	Name	Witness	Time Returned	Name	Witness

REMINDER: Samples must be returned at the end of the shift.

F. SAMPLE/DATA ACCESS AND INTERNAL CHAIN-OF-CUSTODY

1. General Policies and Procedures

PACE has implemented standard operating procedures to assure the integrity of samples and data so that they are not degraded or disclosed to unauthorized personnel. In order to ensure that this policy is maintained, the laboratory facilities are under controlled access. Only employees are allowed into the laboratory facilities; visitors must register at the front desk.

Samples are removed from their proper location by designated personnel and returned to the storage area immediately after the required sample quantity has been taken. This minimizes unnecessary time spent searching for samples and helps prevent matrix degradation from prolonged exposure to room temperature. After the final report is sent and clients are allowed adequate time to review the results, the samples are properly discarded or returned to the client.

PACE normally completes the sample analysis within 15 working days after receipt. Holding times may require faster turnaround times.

Upon client request, additional and more rigorous chain-of-custody protocols for samples and data can be implemented. For samples involving a high degree of confidentiality or potential litigation, PACE, Inc. has developed extensive sample and data handling protocols to assure the scientific and legal defensibility of the report submitted. These protocols include those specified by the USEPA Contract Laboratory Program.

Analysts and technicians follow strict internal chain-of-custody procedures to further ensure the validity of all data. All samples are signed out in a sample custody log book when they are removed for analysis. The sample ID, date, time, analyst, and lab of analysis is recorded in the sample custody log (Exhibit 8) or equivalent. Samples are signed back in noting date, time, and storage location, upon return.

c. Hazardous Materials

Pure product or potentially heavily contaminated samples are tagged as "hazardous" and stored within a secured area, separate from other samples. This area is used only for hazardous samples and is labeled per OHSA requirements.

d. Special Projects

- Volatiles

  Samples within a project are stored in sample number order in vial containers. The holders are then stored as space permits in the Special Project VOA refrigerated storage area.

e. Asbestos

No refrigeration required. Samples are taken to asbestos lab for storage.

5. Responsibilities for Sample Storage

a. QC Department Manager/Sample Management Officer has direct responsibility for ensuring that the SOP is followed, samples are stored properly upon receipt, and refrigerated storage area temperatures are maintained.

b. Sample custodians are responsible for storing all samples upon receipt into the appropriate storage area, maintaining high level security for those samples under custody, and for keeping a current custody sample inventory.

c. Analytical personnel have the responsibility of daily sample storage area maintenance, disposal of old samples, and providing space for incoming samples in routine storage areas.

d. Assigned individuals are responsible for maintaining and documenting: (a) refrigerated storage area temperatures, and (b) corrective actions.

D. SAMPLE STORAGE

1. General Procedures

Samples for analysis are properly stored in the lab according to container type, preservative, and type of security required by the project.

Samples are stored immediately upon receipt to prevent sample degradation.

2. Refrigerated Storage Area Maintenance

All refrigerated storage areas are maintained at 1°- 4°C. The temperature is monitored and recorded daily. If the temperature falls outside the limit of 1°- 4°C, corrective action is to be taken as follows and appropriately documented.

a. Temperature is monitored at 30 minute intervals with the refrigerator door closed.

b. QC Manager is notified if the problem persists longer than one hour.

c. Samples are relocated to a proper storage environment if temperature cannot be maintained after corrective actions are implemented.

3. Routine Sample Storage

a. General Samples

Samples within each project are stored in sample number order. Waters and soils are generally stored on labeled separate shelves.

4. Specific Procedures

a. Volatiles

Samples within a project are stored in numerical order in vial containers. The holders are then stored where space permits in one of the designated volatile organic refrigerated storage areas.

b. Semi-Volatiles

Samples within a project are stored in numerical order in a designated, refrigerated storage area.

2. When Samples Are Received With No Paperwork

a. If delivered by a client: Client is asked if previous arrangements were made for analysis (and with whom). The client completes a chain-of-custody and/or request for analysis, relinquishes samples to sample custodian personnel, and is given a copy of the C-O-C.

b. If received by courier or shipping:

1st: Routine Client File is checked
2nd: Anticipate Sample Alert File is checked
3rd: Sampling Kit Request File is checked
4th: PACE key client contact is consulted
5th: QC department manager is consulted to determine the designated PACE project manager
6th: Information is requested from the PACE project manager.

c. If analysis information can not be determined on the day of sample receipt, sample data entry personnel proceed to assign sample numbers and put samples on hold. Follow-up with project manager occurs until the analyses are determined and samples can be properly logged in.

3. Responsibilities for Sample Log In

a. Quality Control Manager/Sample Management Officer

- Has the overall responsibility for ensuring that this procedure is implemented for all samples received into the laboratory.
- Has overall responsibility for ensuring that samples are logged in correctly (given that appropriate information has been supplied).

b. Sample Custodian

- Has the primary responsibility of ensuring that sample information is input into the LDMS as described in the SOP.
- Has the responsibility to make recommendations to the QC manager for revising the SOP.

C. SAMPLE LOG-IN

1. General Policies

a. Upon completing sample receipt/custody procedures, all sample and analysis data must be complete and documented on the chain-of-custody or accompanying forms for input into the Lab Data Management System (LDMS).

Sample and analysis data must include:

1. Client name and contact
2. Client number
3. PACE project number
4. PACE project manager
5. Sample descriptions
6. Due date
7. List of analyses requested

b. Sample and requested analyses data are input into the LDMS.

c. All samples received are logged into the LDMS on the day of receipt.

d. A Sample and Analysis Data Entry Form (SADEF) is generated immediately by the LDMS.

Distribution of SADEF:

- To the PACE Project Manager with a photocopy of the chain-of-custody. (Include a copy of the Discrepancy Report is applicable).
- To the QC project file with the original chain-of-custody.
- Photocopy to the Organic or Inorganic Department Manager as it applies for RUSH samples.
- To the client.

e. SADEF is to be reviewed against the chain-of-custody.

f. Sample containers are labeled with the corresponding sample number and the stamped date of receipt.

g. Samples are ready for storage.

EXHIBIT 7

PACE, INC.
DISCREPANCY REPORT FORM

Urgency Level: 1(_____) Requires immediate attention
2(_____) Requires attention today
3(_____) Requires attention this week

Initiator: _____ Client: _____
Date: _____
Project # _____

Sample(s) # _____ _____ _____ _____ _____

_____ _____ _____ _____ _____

_____ _____ _____ _____ _____

Discrepancy (if more space needed, use the back of this form): _____

_____

_____

To QC Manager: _____ Date: _____

Client Notified? YES ( ) NO ( ) Date & Time: _____

Project Manager Notified? YES ( ) NO ( ) Date & Time: _____

QC Response: _____

_____

_____

Project Manager Response: _____

_____

_____

Cause and Resolution (proposed or carried out): Completed by: _____

_____

_____

_____

Manager's Initials:

PM Signature: _____ Date: _____

QC Signature: _____ Date: _____

cc: Project File

EXHIBIT 6

SAMPLE I.D. AND CONDITION FORM

Client: _____
Project No.: _____
Date Received: _____

SAMPLE CONDITION UPON RECEIPT CHECKLIST

Complete checklist (A) during sample receipt. If any items are marked "NO," complete section (B) of this form. Otherwise, go to record samples.

			YES	NO
(A)	1.	Are there custody seals or tapes on the shipping container?	___	___
	2.	Are custody seals on the shipping container intact?	___	___
	3.	Is there a completed Chain-Of-Custody (C-O-C)?	___	___
	4.	Do the numbers of samples received and the sample matrices agree with C-O-C?	___	___
	5.	Are there tags attached to each sample?	___	___
	6.	Are sample tags, sample containers and C-O-C all in agreement?	___	___
	7.	Is the C-O-C complete with requested analyses?	___	___
	8.	Are the samples preserved correctly?	___	___
	9.	Is there enough sample to do all analyses?	___	___
	10.	Do the samples have the proper temperature?	___	___
	11.	Are the sample containers intact (e.g., not broken, leaking)?	___	___
	12.	Are VOA vials head-space free?	___	___
	13.	Are all samples within the holding times for requested analyses?	___	___
	14.	Is pH recorded for non-VOA's?	___	___

(B) Explain "NO" item here: _____
_____
_____

Send a copy of this form to Project Manager with Discrepancy Report Form. Copy of both forms remain in the QC file.

Custodian Signature: _____

PACE INCORPORATED
THE ASSURANCE OF QUALITY

EXHIBIT 5

CHAIN-OF-CUSTODY RECORD
Analytical Request

Client

Address

Phone

Report To:

Bill To:

P.O. # / Billing Reference

Project Name / No.

Pace Client No.

Pace Project Manager

Pace Project No.

*Requested Due Date:

Sampled By (PRINT)

Sampler Signature

Date Sampled

ITEM NO.	SAMPLE DESCRIPTION	TIME	MATRIX	PACE NO.	NO. OF CONTAINERS	PRESERVATIVES: UNPRESERVED	H_2SO_4	HNO_3	VOA			ANALYSES REQUEST	REMARKS
1													
2													
3													
4													
5													
6													
7													
8													

COOLER NOS.	BAILERS	SHIPMENT METHOD OUT / DATE	SHIPMENT METHOD RETURNED / DATE

Additional Comments

ITEM NUMBER	RELINQUISHED BY / AFFILIATION	ACCEPTED BY / AFFILIATION	DATE	TIME

29

ORIGINAL

VII. SAMPLE CUSTODY

A. SAMPLE RECEIPT

Sample shipments are received at the sample receiving area. Sample custodians verify the number of shipping containers received against the numbers listed on the shipping manifest/chain-of-custody. Any damage to the shipping containers or other discrepancy observed is noted on the chain-of-custody before signing it. A copy is kept for future reference.

The external chain-of-custody must be signed by the carrier for relinquishment of samples and signed by sample custodian personnel for sample receipt. The actual chain-of-custody may be supplied by PACE, (Exhibit 5), or may be the client's own form. The chain-of-custody remains in the project file at all times.

B. SAMPLE VERIFICATION

Upon arrival of a sample shipment, sample control personnel perform sample inspection. PACE's Sample I.D. and Condition Sheet (or equivalent) (Exhibit 6) serves as a check-off list of procedures to follow and as documentation of the following:

1. Presence/absence of custody seals or tapes of the shipping containers and the condition of the seals (i.e., intact, broken).

2. Presence/absence of chain-of-custody; (if present, is it complete?)

3. Presence/absence of sample tags; (if present, are they removable?)

4. Agreement/non-agreement between the sample tags, chain-of-custody, and any client documentation.

5. Condition of the samples when received, including:

   - Sample temperature
   - Intact, broken/leaking
   - Headspace in VOA vials
   - Sample holding time
   - Sample pH when required

   If discrepancies are found, the PACE project manager is contacted immediately (verbally and by using a Discrepancy Report Form) (Exhibit 7). If the project manager is not available, the QC manager is contacted for further directions. A copy of a Discrepancy Report Form is attached to the project data package.

C. SAMPLING PROCEDURES FOR SOILS AND SEDIMENTS

Soil and sediments are collected according to procedures in the latest edition of Test Methods for Evaluating Solid Waste, EPA-SW-846.

Soil sampling is designed to determine the depth and range of contamination from spillage or the leaching effects of rain on materials stored above ground. If borings are required, the depth and placement of the borings are planned by the project manager/subcontractor and client, using the suspected range of contamination as a guide.

EXHIBIT 4

FIELD LOG DATA SHEET
PACE, Inc.
WELL SAMPLING

Client:_____ Project:_____ Project #:_____

Sample Site:_____

Well Identification and Description: (Locked_____ Not Locked_____) Key#:_____

ID inches_____PVC:_____Steel:_____Stainless Steel:_____Other:_____Labeled:_____

Total Well Depth (from top of casing)_____meters_____feet Elevation:_____feet

Static Water Level (from top of casing) Before Prepumping:_____meters_____feet

Static Water Level (from top of casing) At Time of Sampling:_____meters _____feet

Static Water Elevation:_____feet Water Column:_____feet One Casing Volume_____gal

Date Prepumped:_____Time Prepumped:_____Volume Prepumped:_____gal

Prepumping Method Used:_____ Pump Rate:_____gpm

Date Sampled:_____Time Sampled:_____Sampling Equipment Used:_____

Sample Temperature:_____°C Sample pH:_____Sample Specific Conductance:_____umho/cm2

Field Measurements Temperature Corrected: Yes___No___Metals Filtered in Field: Yes___No__

Weather Conditions:_____

Observations:_____

_____

Sample Description:_____

Name and Affiliation of Sampler(s)_____

Name and Affiliation of Inspector(s) Present:_____

STABILIZATION TEST

Time	pH	Specific Conductance (umhos/cm2)	Temp. (°C)	Cumulative Volume Removed (gallons)

B. SAMPLING PROCEDURE FOR GROUNDWATER AND SURFACE WATER

Groundwater and surface water sampling techniques employed by PACE are in accordance with the EPA Regional IV Standard Operating Procedures and Quality Assurance Manual, and the PACE Field Services SOP Manual.

Trained field sampling crews are dispatched to the site for sample collection and deliver collected samples to the laboratory.

For groundwater sampling, the water level within the well is determined prior to sampling using an electronic water level meter, then recorded on the field log data sheet with all additional pertinent information (Exhibit 4). The volume of water in the casing is calculated and three to five times that volume is purged from the well. In all cases, the well is purged until the conductivity, temperature, and pH have all stabilized, or the well has been purged dry.

Samples from monitoring wells are taken with a precleaned Teflon or stainless steel bailer. Bailers are precleaned by washing first with detergent, then rinsed with tap water, triple rinsed with deionized water, and baked at 105 C for one hour. Precleaned bailers are used between each sampling point.

All samples collected for metals analysis are preserved with nitric acid. The bailer to be used for sampling is used for purging two inch diameter wells and a gas-driven centrifugal pump is used when larger volumes of water need to be removed (static water levels of less than 25 feet). Wells with static water levels greater than 25 feet and casing diameters greater than 3 inches are purged using a submersible pump.

Quality Control Protocols:

A. All Quality Control (QC) procedures are as specifically required by the method, state, or project requirements.

B. The USEPA requires as a minimum one matrix spike, one duplicate or MSD, one blank, per set of samples of similar matrix with a maximum of 20 samples per set. This is a recommended minimum frequency for QC, unless stated otherwise by method, state or project requirements. A client may also request more frequent QC in which case it will be necessary to collect additional samples.

TABLE 1 (CONT.)

ommon Metals Analysis

	Solid	Water
Sample Container:	Plastic or glass	Plastic or glass
Preservative:	4°C	HNO_3 pH < 2
Preferred Amt.:	100 grams	Preferred Volume: 100 ml
EPA Holding Time:	6 Months	EPA Holding Time: 6 Months

	FLAME**		FURNACE	
Parameter	EPA or Standard Method	SW-846 Method	EPA or Standard Method	SW-846 Method
...timum	202.1	7020	202.2	NA
...mony	204.1	7040	204.2	7041
Arsenic	206.3**	7060	206.2	7061
...ium	208.1	7080	208.2	NA
...yllium	210.1	7090	210.2	7091
Cadmium	213.1	7130	213.2	7131
...cium	215.1	7140	NA	NA
...omium, Total	216.1	7190	218.2	7191
...omium, Hexavalent	Standard Method 312B	7195-7198	218.5	NA
...balt	219.1	7200	219.2	7201
[redacted]	220.1	7210	220.2	NA
	231.1	NA	231.2	NA
...er, Total	236.1	7380	236.2	NA
...]	239.1	7420	239.2	7421
...ium	Standard Method 317B	NA	NA	NA
Magnesium	242.1	7450	NA	NA
...nganese	243.1	7460	243.2	NA
...cury (Cold Vapor)	245.1	7470/7471	NA	NA
...ybdenum	246.1	7480	246.2	7481
...kel	249.1	7520	249.2	NA
...ssium	258.1	7610	NA	NA
...nium	270.3**	7740	270.2	7741
...icon	Standard Method 303C	NA	NA	NA
...er	272.1	7760	272.2	NA
...ium	273.1	7770	NA	NA
...conium	Standard Method 303A	NA	NA	NA
...dium	Standard Method 303A	NA	Standard Method 304	NA
...ium	279.1	7840	279.2	7841
	282.1	7870	282.2	NA
...annum	283.1	NA	283.2	NA
...dium	286.1	7910	286.2	7911
	289.1	7950	289.2	NA

...tals by Inductively Coupled Plasma (ICP): Al, Sb, As, Ba, Be, B, Cd, Ca, Cr, Co, Cu, Fe, Pb, Mg, Mn, Mo, Ni, K, Se, Si, Ag, Na, Tl, V, Zn: EPA ICP Method 200.7 or SW-846 Method 6010

...me A.A. or Hydride

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Organic Analytical Guide

TABLE 1 (CONT.)

Water and Wastewater Analysis

EPA Method	Parameter	Technique	Sample Preparation	Sample Container/ Preservative	Preferred Volume (ml)	EPA Holding Time
601	Purgeable Halocarbons	GC-HALL	P&T	VOA/4°C	40	14 Days
602	Purgeable Aromatics	GC-PID	P&T	VOA/4°C	40	14 Days
603	Acrolein and Acrylonitrile	GC-FID	P&T	VOA/4°C, pH Adjusted	40	14 Days
604	Phenols	GC-FID	EXT	GA/4°C	1000	7/40 Days
605	Benzidines	HPLC-Electrochem	EXT	GA/4°C	1000	7/40 Days
606	Phthalate Esters	GC-ECD	EXT	GA/4°C	1000	7/40 Days
607	Nitrosamines	GC-NPD	EXT	GA/4°C	1000	7/40 Days
608	Organochlorine Pesticides and PCB's	GC-ECD	EXT	GA/4°C	1000	7/40 Days
609	Nitroaromatics and Isophorone	GC-FID + ECD	EXT	GA/4°C	1000	7/40 Days
610	Polynuclear Aromatic Hydrocarbons	HPLC-UV/Fluor or GC-FID	EXT	GA/4°C	1000	7/40 Days
611	Haloethers	GC-HALL	EXT	GA/4°C	1000	7/40 Days
612	Chlorinated Hydrocarbons	GC-ECD	EXT	GA/4°C	1000	7/40 Days
613	2, 3, 7, 8-Tetrachlorodibenzo-p-dioxin	GC/MS	EXT	GA/4°C	1000	7/40 Days
614	Organophosphorus Pesticides	GC-FPD or NPD	EXT	GA/4°C	1000	7/40 Days
615	Chlorinated Herbicides	GC-ECD or Hall	EXT	GA/4°C	1000	7/40 Days
624	Purgeables	GC/MS	P&T	VOA/4°C	40	14 Days
625	Base/Neutrals, Acids and Pesticides	GC/MS	EXT	GA/4°C	1000	7/40 Days

Solid Waste Analysis

EPA Method	Parameter	Technique	Sample Preparation	Sample Container/ Preservative	Preferred Volume	EPA Holding Time
8010	Purgeables Halogenated Volatile Organics	GC-HALL	5030	VOA/4°C	*	14 Days
8015	Purgeables Non-Halogenated Volatile Organics	GC-FID	5030	VOA/4°C	*	14 Days
8020	Aromatic Volatile Organics	GC-PID	5030	VOA/4°C	*	14 Days
8030	Acrolein, Acrylonitrile, Acetonitrile	GC-FID	5030	VOA/4°C	*	14 Days
8040	Phenols	GC-FID	3550	GA/4°C	*	14 Days or 7/40 Days**
8060	Phthalate Esters	GC-ECD	3550	GA/4°C	*	14 Days or 7/40 Days**
8080	Organochlorine Pesticides and PCB's	GC-ECD	3550	GA/4°C	*	14 Days or 7/40 Days**
8090	Nitroaromatics and Cyclic Ketones	GC-FID or ECD	3550	GA/4°C	*	14 Days or 7/40 Days**
8100	Polynuclear Aromatic Hydrocarbons	GC-FID	3550	GA/4°C	*	14 Days or 7/40 Days**
8120	Chlorinated Hydrocarbons	GC-ECD	3550	GA/4°C	*	14 Days or 7/40 Days**
8140	Organophosphorus Pesticides	GC-FPD or NPD	3550	GA/4°C	*	14 Days or 7/40 Days**
8150	Chlorinated Herbicides	GC-ECD or HALL	3550	GA/4°C	*	14 Days or 7/40 Days**
8240	Volatile Organics	GC/MS	5030	VOA/4°C	*	14 Days
8250	Semi-Volatile Organics	GC/MS	3550	GA/4°C	*	14 Days or 7/40 Days**

Technique
Instrument:
- GC Gas Chromatograph
- GC/MS Gas Chromatograph Mass Spectrometer
- HPLC High Performance Liquid Chromatograph

Detectors:
- ECD Electron Capture
- Fluor Fluorescence
- FID Flame Ionization
- FPD Flame Photometric
- HALL Electrolytic Conductivity
- NPD Nitrogen Phosphorous
- PID Photoionization
- UV Ultraviolet

Sample Preparation Method Used:
- EXT Extraction Methods that could be used include 3510, 3520, 3540 and 3550.
- P&T Purge and Trap

- 3510 Separatory Funnel Extraction of Liquid Samples
- 3520 Continuous Liquid-Liquid Extraction
- 3540 Soxhlet Extraction of Solid Samples
- 3550 Sonication Extraction of Solid Samples

- 5030 Purge and Trap. Direct Injection of Liquid Samples. Solid Samples Metoli then Injected.

Sample Container/Preferred Volume:
- GA Glass Amber Bottle with Teflon Lined Cap
- VOA Volatile Organic Analysis 40 ml Amber Glass Vial with Teflon Septum
- * Contact Laboratory for Recommendation

EPA Holding Time:
- 7/40 7 Days for Extraction and 40 Days for Analysis
- ** Depends upon Sample Matrix

NOTE:
The methods shown are those commonly employed in performing environmental analyses. It is not intended to be inclusive of all possible EPA analytical methods or to indicate that any laboratory routinely provides the methods or parameters shown.

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Inorganic Analytical Guide

TABLE 1

Common Non-Metals Analysis

Parameter	Typical Method(s)	Comparable SW-846 Method(s), If Applicable	Sample Container/ Preservative*	Preferred Volume (ml)*	EPA Holding Time*
Acidity	EPA 305.1		P, G/4°C	100	14 Days
Alkalinity	EPA 310.1/310.2		P, G/4°C	100	14 Days
Bacteria, Total Coliform	Standard Method 909A	9131/9132	WK/4°C	100	6 Hours
Bacteria, Fecal Coliform	Standard Method 909C		WK/4°C	100	6 Hours
Bacteria, Total Plate	Standard Method 907		WK/4°C	100	48 Hours
BOD, 5 Day	EPA 405.1		P, G/4°C	500	48 Hours
BOD, 5 Day Carbonaceous	EPA 405.1		P, G/4°C	500	48 Hours
Boron	EPA 212.3		HNO_3< 2	100	6 Months
Bromide	EPA 320.1		P, G	200	28 Days
COD	EPA 410.1/410.2		P, G/4°C, H_2SO_4	250	28 Days
Color	EPA 110.3		P, G/4°C	250	48 Hours
Chloride	EPA 325.2/325.3	9251/9252	P, G	100	28 Days
Chlorine, Residual	EPA 330.1		P, G	500	immed.
Cyanide, Total	EPA 335.2	9010	P, G/4°C, NaOH pH > 12	500	14 Days
Fluoride, Total	Standard Method 413A		P	500	28 Days
Fluoride, Electrode	EPA 340.2		P	200	28 Days
Fluoride, (SPADNS)	EPA 340.1		P	500	28 Days
Grease & Oil	EPA 413.1	9070/9071	G/4°C, H_2SO_4	1500	28 Days
Hardness, Total ($CaCO_3$)	EPA 130.2		P, G/4°C	250	6 Months
Ion Chromatography (including common anions such as: Br^-, Cl^-, F^-, NO_3^-, NO_2^-, PO_4^{3-}, $SO_4^{=}$, $SO_3^{=}$, & others)	EPA 300		P, G/4°C	100	48 hrs.
Nitrogen, Ammonia	EPA 350.1/350.2		P, G/4°C, H_2SO_4	500	28 Days
Nitrogen, Kjeldahl	EPA 351.2/351.3		P, G/4°C, H_2SO_4	1000	28 Days
Nitrogen, Nitrate	EPA 353.2	9200	P, G/4°C	100	48 Hours
Nitrogen, Nitrite	EPA 353.2		P, G/4°C	100	48 Hours
Nitrogen, Nitrate & Nitrite	EPA 353.2		P, G/4°C, H_2SO_4	100	28 Days
Nitrogen, Organic	EPA 351.3		P, G/4°C, H_2SO_4	100	28 Days
Odor	EPA 140.1		G/4°C	1000	24 Hours
Oxygen, Dissolved	EPA 360.1		G - Bottle & Top	500	immed.
pH	EPA 150.1	9040/9041/9045	P, G/4°C	100	immed.
Phenol	EPA 420.1	9065	G/4°C, H_2SO_4	1000	28 Days
Phosphorus, Total	EPA 365.1/365.2		P, G/4°C, H_2SO_4	100	28 Days
Phosphorus, Ortho	EPA 365.1/365.2		P, G/Filter	100	48 Hours
Silica, Dissolved	EPA 370.1		P/4°C	100	28 Days
Solids, Total	EPA 160.3		P, G/4°C	100	7 Days
Solids, Total Volatile	EPA 160.4		P, G/4°C	100	7 Days
Solids, Total Dissolved	EPA 160.1		P, G/4°C	100	7 Days
Solids, Total Suspended	EPA 160.2		P, G/4°C	100	7 Days
Solids, Suspended Volatile	Standard Method 209A		P, G/4°C	100	7 Days
Solids, Settleable	EPA 160.5		P, G/4°C	1 Gal.	48 Hours
Specific Conductance	EPA 120.1	9050	P, G/4°C	100	28 Days
Sulfate	EPA 375.4	9036/9038	P, G/4°C	100	28 Days
Sulfide, Total	EPA 376.1	9030	P, G/4°C, NaOH pH > 9, Zn acetate	500	7 Days
Sulfite	EPA 377.1		P, G	500	immed.
Surfactants	EPA 425.1		P, G/4°C	250	48 Hours
Total Organic Carbon	EPA 415.1	9060	P, G/4°C HCl pH < 2	100	28 Days
Total Organic Halogen	EPA 450.1	9020/9021	GA/4°C	500	14 Days
Turbidity	EPA 180.1		P, G/4°C	100	48 Hours

Sample Containers
P Plastic, polyethylene bottle with a polypropylene cap
G Glass
WK Whirl-Pak®
GA Glass, amber bottle with a Teflon® lined cap

Preservatives
H_2SO_4 Sulfuric Acid
HNO_3 Nitric Acid
NaOH Sodium Hydroxide

*Sample container, preferred volume and holding time are for water matrix. Consult laboratory for solid matrix sampling recommendations.

NOTE:
The methods shown are most commonly employed in performing environmental analyses. It is not intended to be inclusive of all possible EPA analytical methods or to indicate that any laboratory routinely provides the methods or parameters shown.

7. Sample Analysis Data Entry Form Tracking for Bottle Prep QC

Forms will be kept in an Outstanding QC file.

a. When a Report of Laboratory Analysis is received for the project, the Sample Analysis Data Entry Form is moved to the Complete QC file.

b. A copy of the Report of Laboratory Analysis is then routed to QC Data Entry and data are entered into the appropriate data base.

c. The data are reviewed by the supervisor of the Bottle Preparation Area and signed off as being certified "clean" if the following criteria are met. All laboratory contaminants shall be at or below the stated detection limit. If this criterion is not met, the bottles are re-cleaned or discarded and another blank analyzed. If criteria are not met, the supervisor of bottle prep works with QA department to discover and rectify the problem in cleaning procedures.

Sample containers, preservatives, and holding times for representative analytical groups are listed in Table 1. Refer to 40CFR 136 for complete information and details.

3. The last three digits are the lot number. They are assigned in sequential order.

4. When the lot code is assigned, it is documented appropriately.

5. The person who prepared the containers initials the Lot Sheet next to the lot code.

One container per lot (or at minimum frequency of 1%) is used to hold a deionized water blank. (ASTM type II) This blank is analyzed to determine the level of contamination in the lot.

- The appropriate analyses are performed for the given container type.
- Use carbon-filtered, deionized water for all blanks.
- Fill all containers, except VOA's, up to the neck of the bottle.
- Fill VOA's such that no bubbles are trapped when the vial is capped.
- Label each blank with the following information:

  Client: PACE, QC

  Sample description: (Lot Code)
  Date Collected:
  Collected by: (Initials)
  Time Collected:
  Analysis: (As indicated for the bottle type)
  Preservative: (Check appropriate preparation)

6. Complete a Chain-of-Custody form to accompany the samples. Client, sample description, time sampled, preservative, analysis: as listed on the bottle label.

   Report to: (Name of container preparation person)

   Project Name: Container QA

   Requested Due Date: Priority 2

   Matrix: H_2O

   Route samples and Chain-of-Custody to Sample Check-in.

6. Sample Container Quality Control and Lot Assignment

a. Bottles of a given type, prepared in one session, constitute a lot.

b. Lot sizes will vary, depending on the demand for a given bottle type.

c. When a lot is prepared, it is assigned an eight character lot code.

1. The first two characters indicate the bottle type.

GN: General Unpreserved
MU: Metals Unfiltered
NT: Nutrients
CN: Cyanide
PH: Phenol
OG: Oil and Grease
SD: Sulfide
GV: GC VOA Water
GC: GC VOA Solid
GL: GC Q-Amber
GS: GC Sm Amber
GM: GC Misc. Refrigerated
HW: Hazardous Waste
OC: Total Organic Carbon
OX: Total Organic Halides
RA: Radiological

A complete listing of codes can be found in Section I of the LDMS User's Manual (Project & Sample Data Entry)

2. The next three digits indicate the bottle size.

125: 125 mL
250: 250 mL
500: 500 mL
000: 1000 mL and one gallon

8. Cap the bottle with a new, Teflon lined cap.

1. Other Container Preparation

1. Polyethylene bottles (125, 250, 500, and 1000 mL) with plastic caps are used to hold samples for general chemistry analysis.

2. Clear glass bottles (125, 500, and 1000 mL) with foil lined caps are used to hold samples with high oil content to be analyzed for general chemistry parameters.

3. Amber glass, small neck bottles (500 mL) with Teflon-lined caps are used to hold samples for total organic halide (TOX) analysis.

5. Procedure: Containers for Soil Samples

a. Volatile Organic Analysis Sample Container Preparation for Soil Samples

1. Wide-mouth, amber or clear glass containers (65 mL-125 mL) with Teflon-lined caps are used to hold samples for volatile organic analysis.

2. The same preparations procedure is used as is used in preparation of VOA containers for aqueous samples except no preservative is added to the containers. (See #4a)

b. Semivolatile Container Preparation

1. Wide-mouth, amber glass jars (250, 500, and 1000 mL) with Teflon-lined caps are used to hold samples for semivolatile analysis.

2. Preparation procedures are identified as those used in preparation of semivolatile containers for aqueous samples. (See #4b)

c. Inorganic Container Preparation

1. Polyethylene bottles (125, 250, 500, and 1000 mL) with plastic caps or wide-mouth clean glass jars (4 oz., 8 oz., or 32 oz.) with teflon-lined caps are used to hold samples for inorganic analysis.

2. If the samples contain a large quantity of oil, clear glass jars (125, 500, and 1000 mL) with foil lined caps are used instead of the polyethylene bottles.

3. Container preparation procedures are identical to those used in preparation of general containers for aqueous samples.

h. Sulfide Container Preparation

1. Polyethylene bottles (250 mL) with plastic caps are used to hold samples for sulfide analysis.

2. Add 0.5 mL of zinc acetate and NaOH (to pH greater than 9) to each container.

3. Attach a white dot sticker to the lid of each prepared container.

i. Total Organic Carbon (TOC) Container Preparation

1. Polyethylene bottles (250 mL) with plastic caps are used to hold samples for TOC analyses.

2. Add 0.25 mL of 1:1 sulfuric acid.

3. Attach an orange dot sticker to each prepared container.

j. Radiological Containers Preparation

1. Polyethylene bottles (one gallon) with wax coated, paper lined caps are used to hold samples for radiological analysis.

2. Add five mL of 1:1 nitric acid to each bottle.

3. Attach a pink dot sticker to the cap of each prepared container.

k. Carbon-Free Deionized (CFDI) Water Container Preparation

1. One gallon, small-mouth, amber glass bottles with Teflon lined caps are used to transport CFDI water.

2. These containers can be reused after appropriate cleaning.

3. Wash the bottle in hot tap water and Acationox detergent, or equivalent, (American Scientific Products).

4. Thrice rinse the bottle with hot tap water.

5. Thrice rinse with CFDI water.

6. Bake the bottle at 103° until dry (at least four hours).

7. Remove the bottle from the oven, cover the mouth with foil, and let cool.

e. Cyanide Container Preparation

1. Polyethylene containers (1000 mL) with plastic caps are used to hold samples for cyanide analysis.

2. Add one gram (8 to 10 pellets) or concentrated solution (1.5-2.0 ml 6N) of sodium hydroxide and one gram of ascorbic acid to each container. If chlorine is present in the sample, use ascorbic acid.

3. Attach a silver dot sticker to the cap of each prepared container.

4. Cyanide containers have a short shelf life; do not prepare in large quantities. (See #6b)

5. Due to a short shelf life, cyanide containers should be prepared as needed.

f. Phenol Container Preparation

1. Clear or amber glass, small mouth containers (1000 mL) with "poly seal" caps are used to hold samples for phenol analysis.

2. Add 1.5 - 2.0 mL of sulfuric acid, diluted 1:1 from concentrate with carbon-filtered deionized water, to each container.

3. Attach an orange dot sticker to the cap of each prepared container.

g. Oil and Grease Container Preparation

1. Clear or amber glass, wide-mouth containers (1500 mL) with foil lined caps are used to hold samples for oil and grease analysis.

2. 1000 mL amber glass containers with Teflon lined caps are acceptable.

3. Add five mL of 1:1 sulfuric acid to each container.

4. Attach an orange (color-coded) dot sticker to the cap of each prepared container.

c. Metals Container Preparation

1. Polyethylene bottles (125, 250, 500, and 1000 mL) with plastic caps are used to hold water samples to be analyzed for metals.

2. Add a small amount of 1:1 nitric acid to a bottle.

3. Cap the bottle and shake vigorously, being certain the acid comes in contact with all interior surfaces.

4. Empty the container.

5. Rinse the bottle and cap thrice with deionized water.

6. Add the appropriate amount of 1:1 nitric acid, cap, and place a red dot on the cap to indicate the container contains nitric acid preservative.

Container Size	Quantity 1:1 Nitric Acid
125 mL	0.25 mL
250 mL	0.38 - 0.5 mL
500 mL	0.75 - 1.00 mL
1000 mL	1.5 - 2.0 mL

d. Nutrient Container Preparation

1. Polyethylene bottles (250, 500, and 1000 mL) with plastic caps are used to hold water samples for nutrient analysis.

2. Add the appropriate amount of sulfuric acid, diluted 1:1 from concentrate with carbon filtered deionized water, to each container.

Container Size	Quantity 1:1 Sulfuric Acid
250 mL	0.38 - 0.5 mL
500 mL	0.75 - 1.00 mL
1000 mL	1.5 - 2.0 mL

3. Attach an orange dot sticker to the cap of each prepared container.

3. Assembling VOA vials.

   a. Place ten clean vials upright in a vial box with dividers. Recover drying trays with foil after vials have been removed.

   b. Add 4 drops of concentrated hydrochloric acid (HCL).

   c. Add (10 mg/40 ml) 0.008% sodium thiosulfate if chlorine is present (e.g. drinking water).

   d. Assemble a cap by inserting a septum in the cap such that the Teflon (white) side is exposed to the interior of the vial.

   e. Cap each vial tightly.

   f. Repeat assembly procedures until all vials are capped.

b. <u>Semivolatile Container Preparation</u>

   1. Glass, amber jars (250, 500, and 1000 mL) with Teflon lined caps are used to hold samples for semivolatile analysis.

   2. Bottles and cap liners are rinsed with reagent grade acetone. (Acetone is a target compound for some EPA methodologies and a CLP compound. If acetone interferes with the analyses, use of hexane and/or methanol may be an alternative, as specified in the method.)

      a. Acetone is highly flammable and acetone vapors are toxic.

      b. When using acetone, wear latex gloves, safety glasses and work in a vented hood.

      c. Pour a small amount of reagent grade acetone in the bottle to be rinsed.

      d. Cap the bottle with a Teflon lined cap.

      e. Shake the bottle making sure the acetone comes in contact with all sides of the bottle and the cap liner.

      f. Empty the bottle, invert it on a drying rack and allow it to air dry.

      g. Cap the bottle with a rinsed cap.

      h. Attach a blue dot to the top of the cap indicating the container has been acetone rinsed.

4. Procedures: Containers for Aqueous Samples

a. Volatile Organic (VOA) Sample Container Preparation

1. Vial cleaning procedures.

a. Wash an entire package of vials in one washing session. Never store open packages of vials.

b. Soak the vials in cleaning solution (one capful of Acationox detergent, or equivalent, per sink of hot tap water) for 5 minutes.

c. After soaking, thrice rinse each vial thoroughly with hot tap water.

d. Thrice rinse each vial thoroughly with carbon filtered, deionized water (CFDI).

e. Stack rinsed vials in a drying tray (metal tray lined with aluminum foil, dull side exposed).

f. Bake the vials at 103°C for a minimum of four hours.

g. Cover baked vials with aluminum foil such that the dull side of the foil is in contact with the vials and set trays on a lab bench to cool.

2. Septum and cap cleaning procedures.

a. Clean entire packages of caps and septa. Do not store open bags.

b. Clean caps and septa separately.

c. The same procedures used for vial cleaning are used for cap and septum cleaning. Follow B through D in Section 1.

d. Spread evenly and thinly in drying trays to facilitate drying.

e. Dry for one hour at 103°C. Extended periods of heat can damage caps and septa.

f. Place clean caps and septa into a 1500 mL glass container which has been cleaned.

VI. SAMPLING PROCEDURES

PACE, Inc. receives samples collected by clients and also has the capability to perform sampling for clients. PACE prepares or purchases sample containers in accordance with EPA-issued guidelines for container and preservative requirements. Pre-cleaned containers are obtained from reputable vendors and preservatives are added as required. Technical assistance from all supervisory and management staff is available to clients if needed.

A. BOTTLE PREPARATION PROCEDURES

The following is the procedure used for Sample Container Preparation:

1. Purpose

The purpose of this Standard Operating Procedure (SOP) is to provide clear, consistent methods for preparing containers for sample collection. Following this procedure will facilitate accurate and consistent analytical results.

2. Application

The policies and procedures contained in this SOP are applicable to the personnel in the container preparation area.

3. General Policies

a. Always use new bottles when preparing containers for sampling (exception: One gallon, amber glass bottles used for transporting deionized water can be re-used after proper cleaning). These may be commercially-obtained precleaned bottles.

b. Always wear disposable latex gloves when handling sample containers.

c. Several preparation procedures require the use of acids as a preservative or cleaning agent.

   1. Be extremely careful when working with acids.

   2. Always wear safety glasses and a laboratory coat.

d. Bottle labels will list the preservatives added and the analysis to be performed, minimizing the probability for error.

e. When shipping pre-preserved bottles containing corrosives or oxidizers, consult proper DOT regulations.

Completeness is a measure of all information necessary for a valid scientific study. For completeness, it is expected that the methodology proposed for chemical characterization of the samples collected will provide data meeting QC acceptance criteria following standard laboratory data review and validation for at least 95% of all samples collected. Completeness may also be defined as a comparison of the number of tests successfully completed (with acceptable QC) to the number of tests requested. Discrepancy reports are completed to provide explanation when QC criteria are not met.

Representativeness is a qualitative element that is related to the ability to collect a sample that reflects the characteristics of that part of the environment that is to be assessed. Sample representativeness is dependent on the sampling techniques used and is considered individually for each project. It is specifically addressed in each work plan.

Comparability is also considered during preparation of the work plan. The objective of comparability is to ensure that results of similar activities conducted by different parties are comparable. PACE uses EPA-approved or other methods and procedures to ensure comparability with data from previous or following studies. PACE participates in external and interlaboratory performance evaluation (PE) studies as additional means of establishing comparability in the laboratory.

V. QUALITY ASSURANCE OBJECTIVES

The purpose of the plan is to define procedures for the documentation, evaluation, validation, and reporting of data. The objective is to provide a uniform basis for sampling, sample handling, instrument maintenance and calibration, methods control, performance evaluation and analytical data generation and reporting. Specific procedures to be used for sampling, chain of custody, calibration of field instruments (pH, conductivity meters, etc.), laboratory analysis, reporting, internal quality control, audits, preventive maintenance, and corrective actions are described in specific sections of this plan. This section addresses the objectives of accuracy, precision, completeness, representativeness, and comparability.

The QA objectives for precision and accuracy are to achieve the QC acceptance criteria specified in the proposed analytical procedures. For the organic and inorganic procedures, the precision and accuracy guideline requirements are specified in the individual methods.

Field blanks and duplicates are collected and analyzed to assess field sampling activities. The results check procedural contamination and/or ambient conditions at the site.

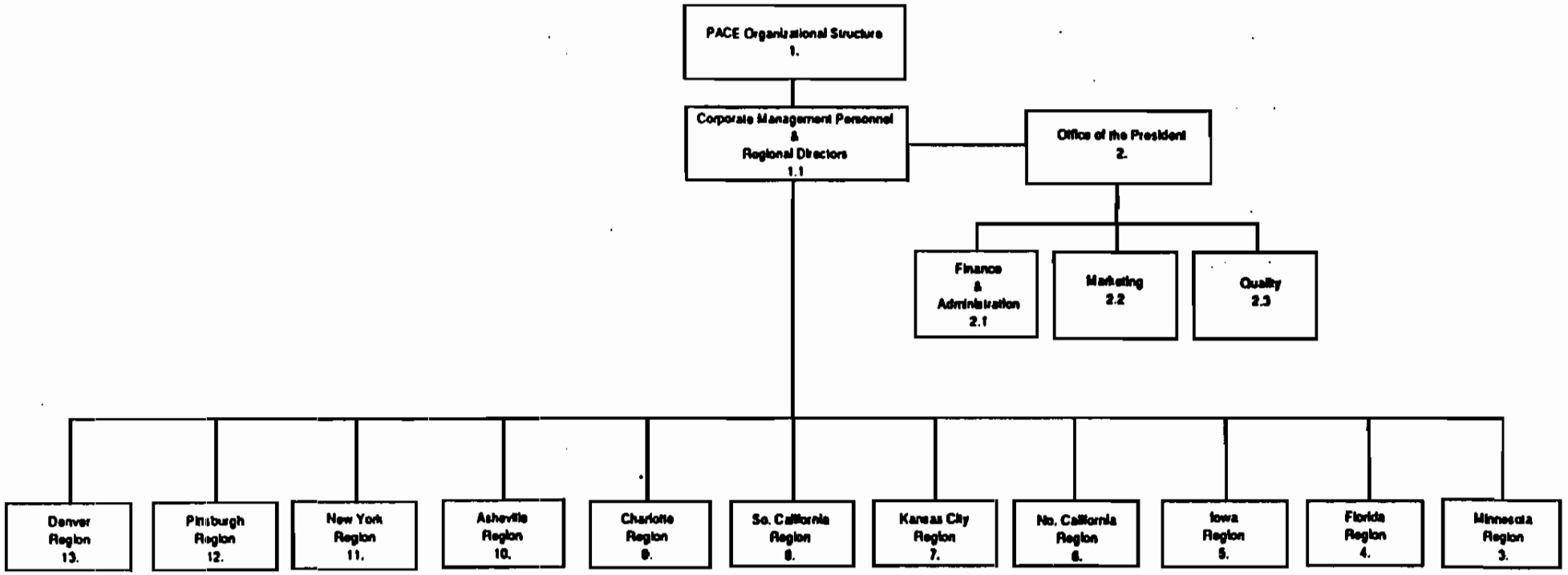
Due to the extensive number of organic parameters and potential matrices, the development of precision and accuracy objectives and control limits for every matrix is difficult. This is typically done with (1) matrix spike and matrix spike duplicate compounds which are added to selected samples before extraction and analysis, and/or (2) surrogate spike compounds which are added to every sample, before extraction and analysis. Although the surrogate and matrix spike analyses do not provide statistically valid statements about precision and accuracy for every compound in a sample, they do give the data reviewer enough information to make judgements about precision and accuracy on a sample-by-sample basis.

Inorganic precision and accuracy data are determined by using duplicate samples (precision), matrix spike and laboratory control samples (accuracy). The following procedure is used:

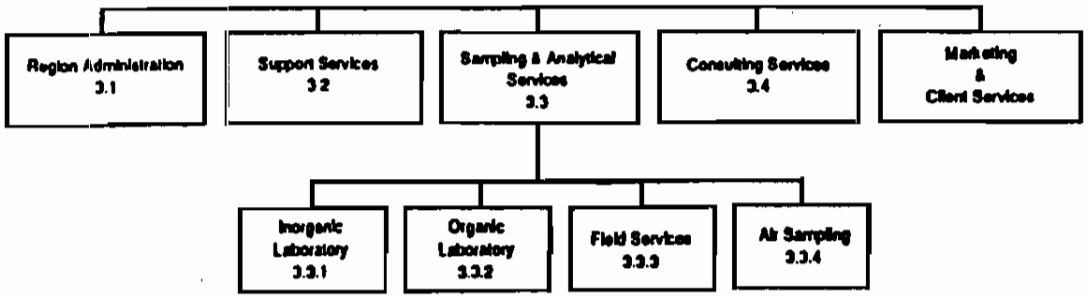
For a duplicate sample analysis, at least one duplicate sample is analyzed per sample matrix type (e.g. water, soil) and concentration (e.g. low, medium) per batch of samples or for each 20 samples received, whichever is more frequent, or as specified by state/project requirements. Samples identified as field blanks can NOT be used for duplicate samples analyses. If two analytical methods are used to obtain the reported values for the same element for a batch of samples (i.e., ICP, GFAA), duplicate samples will be run by each method. The relative percent difference (RPD) for each component is calculated for later use during data assessment.

EXHIBIT 3

Guide To Organizational Charts



These charts detail the MN Region divisions and departments and represent the full model on which all regional office structures are based.



Aug[redacted]91

EXHIBIT

Corporate Management Personnel & Regional Directors

1.1

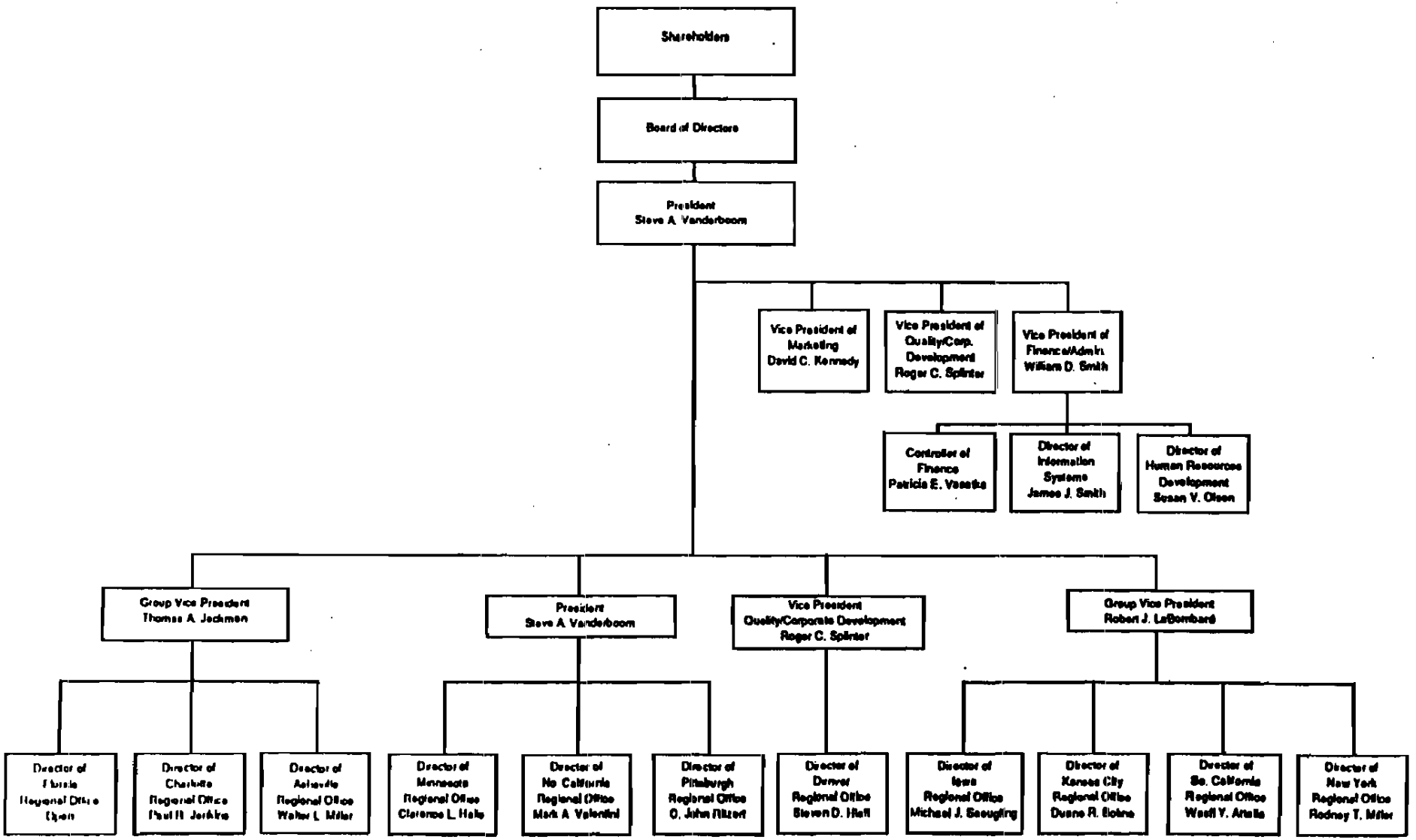
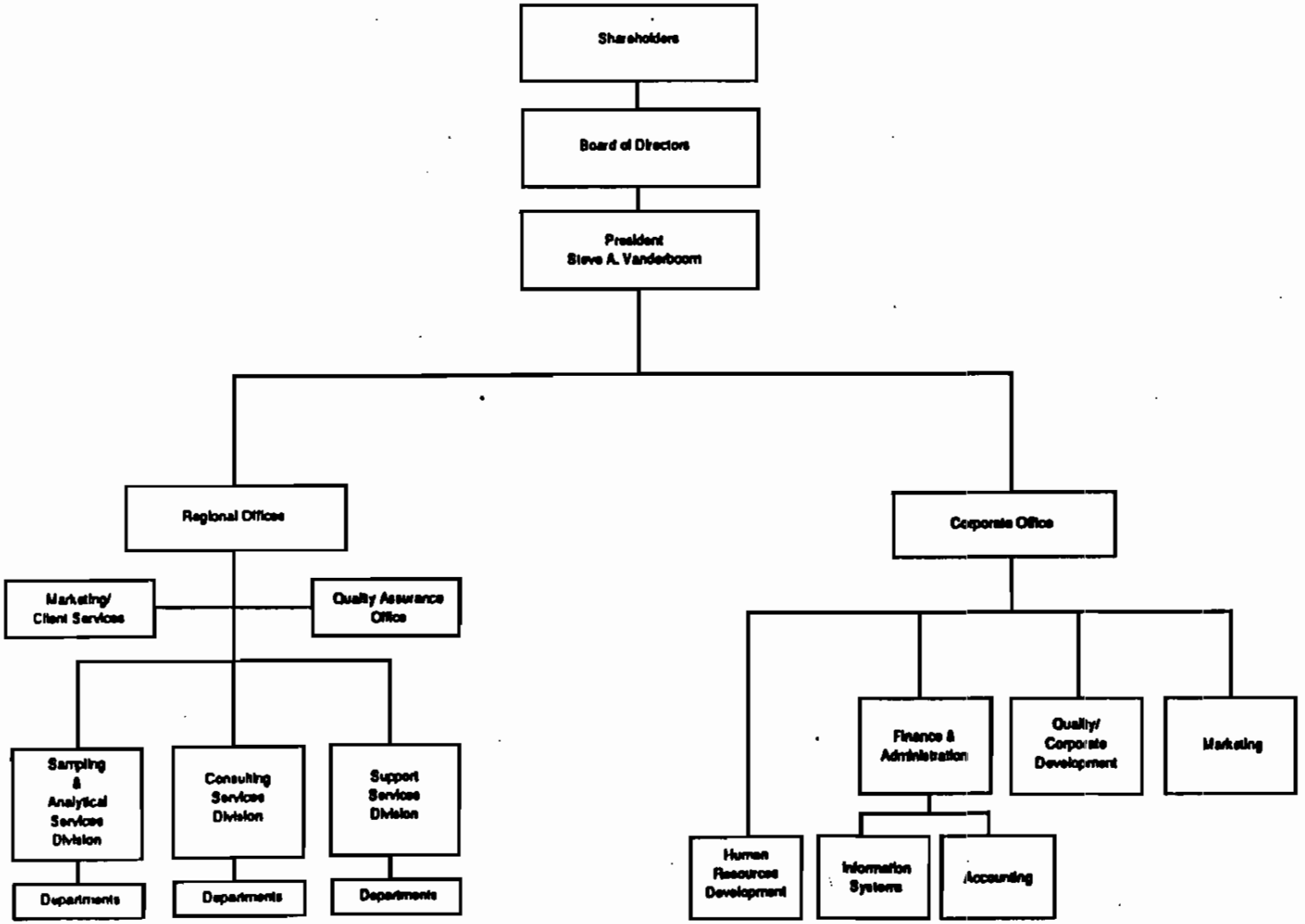


EXHIBIT 1

PACE Organizational Structure



September 1991

IV. LABORATORY ORGANIZATION AND RESPONSIBILITY

The organizational structures for PACE, Inc. are provided in Exhibits 1, 2, and 3.

Exhibit #1	Illustrates the PACE, Inc. Organizational Structure
Exhibit #2	Illustrates the PACE Corporate Structure with Regional Designation
Exhibit #3	Illustrates the full model on which all regional office structures are based

Job descriptions are provided within Quality Assurance Project Plans, as they are designed and developed to address specific projects.

C. STATEMENT OF POLICY

PACE, Inc. is committed to the policy of providing the highest quality product to its client. The validity and reliability of the information generated is maximized by the adherence to documented quality control procedures and quality assurance protocols. PACE emphasizes the application of sound quality assurance/quality control principles beginning with the initial planning of the project, through all the field and laboratory activities and ultimately to the generation of the final report. The principles of data quality objectives, representativeness, completeness, comparability, precision and accuracy are applied.

PACE is committed to providing the resources, including facilities, equipment and personnel, to ensure the adherence to rigorous QA/AC protocols. Individual Quality Assurance Project Plans are developed for monitoring analytical projects to conform with the established QA/QC protocols.

NEW ENGLAND REGION

NH

QUALITY

ASSURANCE

MANUAL

PACE NEW ENGLAND, INC.

Pace NE, Inc.
P.O. Box 2130,
One Lafayette Road
Hampton, NH 03842
(603) 926-7777

ANALYTICAL LABORATORY QUALITY ASSURANCE MANUAL

TITLE: PACE New England, INC. Laboratory Quality Assurance Manual

Prepared By/Date: Stephanie Beck/Wendy E. Harris March 1993

Approved By/Date:

Stephanie Beck 3-22-93
QA Officer Date

[signature] 3-22-93
Regional Director Date

Section	Revision No.	Date
1	2	3/93
2	2	3/93
3	2	3/93
4	2	3/93
5	2	3/93
6	2	3/93
7	2	3/93
8	2	3/93
9	2	3/93
11	2	3/93
12	2	3/93
13	2	3/93
14	2	3/93
15	2	3/93
16	2	3/93

Date of Issue: March 1993

Supersedes No.: ALL

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1.0 INTRODUCTION

1.1 Purpose and Scope

This manual details the quality assurance program in effect in the PACE NE Analytical Chemistry Laboratory. It is meant to be a teaching tool and source of information for laboratory personnel. The Manual is divided into logical sections, each dealing with a different phase of laboratory operation, yet all sections overlap and function together to form a complete quality assurance program. The Manual is based on Good Laboratory Practices, common sense, and industry-accepted standard analytical practices.

The Manual must be read and understood by all laboratory personnel as part of their training program. The Manual should also be referred to regularly as a source of information. A system of continuous updating is built into the Manual to allow it to change as laboratory conditions change or as new regulations are promulgated. This manual is a controlled document, which means that its identity, development, distribution, and status must be known and traceable at all times. All PACE NE permanent laboratory personnel will be assigned a controlled copy.

Whenever a technician or analyst is in doubt as to proper procedures in a specific circumstance, the Manual should be consulted. Omissions or errors should be immediately reported to the Quality Assurance Officer, for corrective action. **IT IS THE RESPONSIBILITY OF EACH LABORATORY WORKER TO ENSURE THAT THE PROVISIONS OF THIS MANUAL ARE FOLLOWED.** Disagreement with specific requirements or knowledge of changes causing deviation from the procedures should be discussed with the immediate supervisor before further work is completed. Laboratory personnel are encouraged to comment on the Manual and make recommendations for more efficient procedures.

The latest revision of each section of the Manual is the applicable rule. Therefore, revisions will be announced to all laboratory personnel. An uncontrolled copy of the Manual is offered to clients and regulatory agencies as the definitive quality assurance program used at PACE NE.

1.2 QA Policy and Objectives of the Program

PACE NE is committed to quality as priority number one. PACE NE's quality assurance policy is based on the definition of quality as conformance to requirements; and further, on the premise that the requirements are governed by Company policies and standard

shall become part of the document control number when the SOP is accepted for implementation by PACE NE management. SOP's shall be reviewed and approved by the Group Supervisor (and Lab Manager for all SOP's related to analytical procedures) and the QA Officer, and submitted by the QA Department to the Technical Directors and the Regional Director for approval prior to implementation.

1.3.3 Project QA Manuals

Project QA Manuals shall be implemented as required. These shall include such documents as Quality Assurance Project Plans (QAPP's). For those projects which require specific QA/QC criteria, a QAPP which has been approved by a regulatory agency, usually the EPA, is provided to PACE NE by the client.

1.3.4 Document Control, Distribution and Revision

In order that this document achieve the goals outlined in Section 1.2, it is necessary that each PACE NE laboratory employee be familiar with the current provisions of this document. It is also necessary that this document represent a consensus among PACE NE Management and operational personnel as to the quality level desired and the means to that end.

Prior to its publication as a controlled document, this manual must be approved by the Quality Assurance Officer, the Laboratory Technical Directors, and the Regional Director. To obtain such approval, the document proceeds through an iterative process of review and revision, involving the affected managers and their designated representatives. The signature page at the beginning of the manual represents acceptance.

Each time a revision is made to this manual, it must also be approved. The Quality Assurance Officer must approve each revision. If the revision constitutes a complete rewrite of the document, then review and approval by the Quality Assurance Officer, the Laboratory Technical Directors, and the Regional Director becomes necessary. The appropriate approval process will be decided in each case by the Quality Assurance Officer.

operating procedures. This commitment recognizes the need for data to be representative of the environmental conditions under consideration, and for data to be generated within a system of functions that that is designed to meet applicable regulatory compliance criteria. To this end, PACE NE has developed a company-wide Quality Assurance (QA) Plan and maintains an ongoing QA Program. Our Quality Assurance Program contains provisions for establishing, maintaining and executing protocols which lead to results of known, appropriate and acceptable quality; documentation of these activities is an integral part of the QA program. No other concern will be permitted to interfere with the quality of data PACE NE provides to clients.

This manual describes the set of policies and principles which guide day-to-day operations. Specific protocols are included by reference and are contained in a series of volumes cited in Section 8.0 of this document.

1.3 Quality Assurance Documents

1.3.1 QA Manual

This document describes management policies related to operation of the analytical laboratories. It provides overall guidance regarding acceptable practices and discusses each element of the Quality Assurance Program. It functions as the Project QA Manual where no other Quality Assurance Project Plan, Statement of Work or other contractually mandated project plan has been specified. Adherence to the practices described in this manual is required of all employees. This manual may be revised and/or superseded only with the written authority of the PACE NE Regional Director. Copies of this manual are controlled and distribution is administered by the QA Officer.

1.3.2 Standard Operating Procedures Manuals

All procedures related to sample collection, storage, preparation, analysis, disposal, data validation, data reporting and employee training and safety shall be contained in written Standard Operating Procedures Manuals (SOP's). Each SOP shall contain the elements outlined in PACE NE SOP QA-553, Preparation of SOP's. All sections shall be structured in a step-wise manner using numbered sections. All record-keeping requirements shall be described at each step in the SOP. Examples of forms used shall be included as tables or figures and referenced within the text. Preparation of SOP's shall be the responsibility of each Group Supervisor. SOP's shall be assigned a number from the Inventory List for SOP's maintained by the Quality Assurance Department. This number

1.4 Terms and Definitions

Accuracy: The degree of agreement between a measured value and the true or expected value.

Aliquot: A measured portion of a sample taken for analysis.

Analyte: The specific entity an analysis seeks to determine.

Batch: A grouping of samples of similar matrix which are prepared and analyzed together with the same method and the same lots of reagents within the same time frame. A sample may be analyzed in a different analytical batch than the one with which it was prepared.

Blank: A blank is an artificial sample designed to detect and/or monitor the contribution of analyte and non-analyte contamination, instrumental background and sample processing to the measurement system.

Blind Sample: A sample submitted for analysis whose composition is known to the submitter but unknown to the analyst.

Calibration: The process of establishing the relationship between instrument response and known, traceable quantities of analytes of i

Comparability: Comparability is a qualitative parameter expressing the co ence with which one data set can be compared to another. Compa e data are produced through the use of standardized procedures and techniques.

Completeness: Measure of the amount of valid data obtained from a measurement system compared to the amount that was expected to be obtained under correct normal conditions.

Continuing Calibration: The process of analyzing standards periodically to verify the maintenance of calibration of the analytical system.

Control Chart: A graphical plot of test results with respect to time or sequence of measurement, together with limits within which they are expected to lie when the system is in a state of statistical control.

Control Limit:	A range within which specified measurement results must fall to signify compliance. Control limits may be mandatory, requiring corrective action if exceeded, or advisory, requiring that nonconforming data be flagged.
Detection Limit:	The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero.
Dry Weight:	The weight of a sample based on percent solids. The weight after drying in an oven.
Duplicate Analysis:	A second measurement made on the same sample extract or digestion to assist in the evaluation of precision of analysis.
Duplicate Sample:	A second aliquot of the same sample that is treated the same as the original sample in order to determine the precision of the method.
Equipment Blank:	Special type of field blank used primarily as a check on equipment decontamination procedures. Laboratory deionized water is passed over sampling equipment after decontamination.
Field Blank:	A quality control sample used to assess the contamination effects on accuracy due to the combined activities of sampling and analysis. Typically, it is composed of a reagent and analyte free matrix (deionized water) provided by the laboratory.
Field Sample:	A portion of material received by the laboratory to be analyzed, that is contained in single or multiple containers and identified by a unique field ID number.
Holding Time:	The elapsed time expressed in days from the date of sample collection by the field personnel until the date of its processing/analysis. For the Contract Laboratory Program, holding times start at the Verified Time of Sample Receipt by the laboratory. Holding time requirements are dictated by the method or QAPjP.

Homogeneity: The degree to which a property or substance is evenly distributed throughout a material.

Instrument Detection Limit: The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. The instrument detection limit is generally more sensitive than the method detection limit.

Initial Calibration: The process of analyzing standards, prepared at specified concentrations, to define the quantitative response, linearity and dynamic range of the instrument to the analytes of interest. Initial calibration is performed whenever the results of a continuing calibration do not conform to the requirements of the method in use or at a frequency specified in the method.

Internal Standards: Analytes added to every standard blank, matrix spike, matrix spike duplicate, and sample at a known concentration, prior to analysis for the purpose of adjusting the response factor used in quantitating target analytes. Internal standards are used as the basis for quantitation of the target compounds, and are generally applicable to organic analyses.

Lab. Control Sample: A control sample of known composition spiked with known concentration of analytes of interest. Aqueous and solid laboratory control samples are analyzed using the same preparation, reagents, and analytical methods employed for field samples received.

LABUX: The Hewlett-Packard A900 based Laboratory Information Management System. It is used to collect data from instrumentation and preparation laboratories for later combination into final reports. Data is collected a number of ways including direct ports from instrument computers, interactive manual entry direct to the database using HP provided procedures, importing data from spreadsheets, and importing batch data entered manually. Data is combined, calculated, and formatted either by HP provided or custom reporters and stored in directories where it can be printed initially for manager review and later as part of a final report.

LAS:	Hewlett-Packards' A900 based 3350a Laboratory Automation System. Its function is to collect, calculate, and present chromatographic data. The system allows the operator to calibrate for, identify and quantitate the data. Requantitation is possible without reinjection of the sample. Typically the data provided to the analyst by the LAS is entered into LABUX for combination with data from the preparation laboratory to generate a final report. Alternatively, the preparation information can be entered into the analysis sequence on the LAS to generate results corrected for preparation volumes, dilutions, and dry weights.
Lot:	A quantity of bulk material of similar composition processed or manufactured at the same time.
Matrix:	The predominant material of which the sample to be analyzed is composed.
Matrix Spike:	Aliquot of sample fortified (spiked) with known quantities of specified compounds and subjected to the entire procedure in order to indicate the appropriateness of the method for the matrix by measuring recovery.
Matrix Spike Duplicate:	A second aliquot of the sample that is treated the same as the original matrix spike sample. The relative percent difference between the matrix spike and matrix spike duplicate is calculated and used to assess analytical precision.
Method Blank:	An analytical control consisting of all reagents, internal standards and surrogate standards, that is carried through the entire analytical procedure. The method blank is used to define the level of laboratory background, contamination, and variation in the associated sample batch.
Method Detection Limit:	Method Detection Limits may be determined using replicate spike samples The detection limit is calculated using the appropriate student's t-parameter times the standard deviation of a series of spiked samples.
Performance Audit or Evaluation:	A process to evaluate the proficiency of an analyst or laboratory by evaluation of the results obtained on known test materials.

Precision: The measurement of agreement of a set of replicate results among themselves without any prior information as to the true result. Precision is assessed by means of duplicate/replicate sample analysis.

Protocol: A stated plan that clearly defines the objectives, methods and procedures for accomplishing a task.

QAPP: A Quality Assurance Project Plan or QAPP is a project specific document that describes the policies, organization, objectives, functional activities, and specific QA and QC activities designed to achieve the data quality goals of a specific project.

Quality Assurance: A system of policies and procedures whose purpose is to ensure, confirm and document that the product or service rendered fulfills the requirements of PACE NE and its client. Quality Assurance includes quality planning, quality control, quality assessment (auditing), quality reporting and corrective action.

Quality Control: A system of checks and corrective measures, integrated with the activities that directly generate the product or service, that serves to monitor and adjust the process to maintain conformance to predetermined requirements.

Replicate Samples: A second, separate sample collected at the same time, from the same place, for the same analysis, as the original sample in order to determine overall precision.

Reporting Limit: The level at which method, permit, regulatory and client specific objectives are met. The reporting limit may never be lower than the statistically determined MDL, but may be higher based on any of the above considerations. Reporting limits are corrected for sample amounts, including the dry weight of solids, unless otherwise specified.

Rounding Rules: If the figure following those to be retained is less than 5, the figure is dropped, and the retained figures are kept unchanged. As an example,

11.443 is rounded off to 11.44. If the figure following those to be retained is greater than 5, the figure is dropped, and the last retained figure is raised by 1. As an example, 11.446 is rounded off to 11.45. If the figure following those to be retained is 5, and if there are no figures other than zeros beyond the five, the figure 5 is dropped, and the last-place figure retained is increased by one if it is an odd number or it is kept unchanged if an even number. As an example, 11.435 is rounded off to 11.44, while 11.425 is rounded off to 11.42. If a series of multiple operations is to be performed (add, subtract, divide, multiply), all figures are carried through the calculations. Then the final answer is rounded to the proper number of significant figures.

Sensitivity: Capability of methodology or instrumentation to discriminate between samples having differing concentrations or containing differing amounts of an analyte.

Split Sample: A portion or subsample of a total sample obtained in such a manner that is not believed to differ significantly from other portions of the same sample.

Standard: A substance or material the properties of which are believed to be known with sufficient accuracy to permit its use to evaluate the same property in a sample.

Standard Blank: An analytical control sample consisting of the same solvent/reagent matrix used to prepare the calibration standards without the analytes. It is used to construct the calibration curve by establishing instrument background.

Standard Operating Procedure: A procedure adopted for repetitive use when performing specific measurement or sampling operation. It may be an industry accepted standard method or one developed by the user.

Surrogates: When employed, these are compounds added to every blank, sample, matrix spike, matrix spike duplicate, and standard prior to any processing or preparation; used to evaluate analytical efficiency by measuring recovery. Surrogate compounds are not expected to be detected in environmental media, but are similar to the analytes of interest. Surrogates are generally utilized for organic analyses.

Systems Audit: An on-site inspection or assessment of a laboratories' quality control system.

Traceability: The ability to trace the source and accuracy of a material (i.e. standard) to a recognized primary reference source such as the National Institute of Standards and Technology (NIST) or USEPA. Also, the ability to independently reconstruct and review all aspects of the measurement system through available laboratory notebooks and documentation and reach the same results.

Trip Blank: This blank is used to detect sample contamination from the container and preservative during transport and storage of the sample. A cleaned sample container is filled with laboratory pure water; any preservative used in the sample is added; and then the blank is stored, shipped, and analyzed with its group of samples.

Validation: The process by which a sample, measurement, method, or piece of data is deemed useful for a specified purpose.

[illegible] Limits The limits (typically 2 standard deviations either side of the mean) shown on a control chart within which most results are expected to lie (within a 95% probability) while the system remains in a state of statistical control.

2.0 QA ORGANIZATION AND PERSONNEL

It is important for efficient laboratory operation that all laboratory employees understand the operational structure, specific areas of responsibility and lines of authority within the organization.

It is equally important for laboratory personnel to understand that the structures of the Quality Organization may be separate from other laboratory operations but that the quality function is totally integrated into every aspect of laboratory operation. All laboratory personnel are responsible for knowing and following proper methods and standard operating procedures; recording quality control information required by those procedures in the proper location; and suspending analyses when quality control criteria are not met.

The organizational structure of the analytical chemistry laboratory is provided in Figure 2-1. Under the direction of the laboratory technical directors, the technical staff of the laboratory is organized into the following functional groups:

Sample Management (Receipt and Log-in)
Sample Preparation - Organic
Sample Preparation - Metals
Wet Chemistry
Metals Analysis
GC Analysis
GC/MS Volatiles Analysis
GC/MS Semivolatiles Analysis
Reporting
Project Management (Client Services)

Each group is headed by a Group Supervisor who is responsible for operations on a daily basis. Environmental chemists, analysts, laboratory technicians and laboratory assistants report to the Group Supervisors. See Appendix A for resumes of all technical staff employed as of this revision date.

2.1 Laboratory Organization

It is the individual responsibility of each analyst and technician to perform their assigned tasks according to the applicable SOPs, QA Project Plans, Study Protocols, and Work Plans. This includes responsibility for performing quality control analyses as specified in the method SOP and for entering the QC data in the appropriate method control file

system. The analyst shall report out-of-control results to the Group Supervisor.

Group Supervisors shall assure that analysts and technicians are instructed in the requirements of the PACE NE Laboratory QA Manual, study-specific QA Project Plans, SOPs, Protocols, and Work Plans for the analytical method or other procedure. Group Supervisors shall review sample QC data at frequent intervals designed to assure that QC analyses are being performed at the required frequency, that data are documented in the method control file system and that established corrective procedures for out-of-control situations are followed and the results documented. It is the responsibility of the Group Supervisor to assure that data have been validated and reported to the Laboratory Manager. Group Supervisors shall report to Laboratory Managers.

Laboratory Managers shall be the Technical Directors and shall take overall responsibility for technical conduct, evaluation and reporting of all analytical tasks associated with each study. Laboratory Managers assure that approved procedures are documented and followed, that all data are recorded and verified and that all deviations from approved procedures are documented. Laboratory Managers shall assure that Group Supervisors are instructed in the requirements of the PACE NE Laboratory QA Manual, study-specific QA Project Plans, SOPs, Protocols, and Work Plans. Laboratory Managers approve standards for QC control limits for methods and work with Group Supervisors to bring out-of-control methods back to within established acceptance limits. Laboratory Managers shall report to the Regional Director.

The Quality Assurance Department, under the direction of the Quality Assurance Officer, shall be responsible for conducting systems audits and inspections for compliance with this manual, SOP's and QA Project Plans or other project-specific protocols, maintaining the archives, maintaining historical files of all QA documents, reviewing QC charts, documenting findings and corrective actions, and reporting findings to management. The Quality Assurance Officer shall report directly to the Regional Director of PACE NE.

The PACE NE Regional Director shall designate Laboratory Manager(s)/Technical Director(s) and replace if necessary. The PACE NE Regional Director shall assure that there is a Quality Assurance Department, that personnel and other resources are adequate, that personnel have been informed of their responsibilities, that deficiencies are reported to Laboratory Managers and that corrective actions are taken and documented. Any significant changes to written SOP's shall be authorized in writing by the Regional Director of PACE NE.

2.2 Training and Orientation

Each new permanent employee receives a four part orientation: a human resources orientation, a safety department orientation, a quality assurance department orientation, and a supervisory orientation. The human resources orientation involves matters of immediate personal concern such as benefits, salary, and company policies. The safety department orientation is an in-depth examination of the PACE NE Chemical Hygiene Plan and safety program, which are consistent with the requirements of OSHA's Hazard Communication Program (29 CFR 1910.1200). The Quality Assurance orientation provides the new employee with information on the PACE NE QA program through a brief introduction to the QA manual and SOPs, acceptable recordkeeping practices, and the individuals responsibility. The new employee's group supervisor provides the employee with a basic understanding of the role of the laboratory within the structure of PACE, Inc. and the basic elements of that individual's position within the laboratory. The training of a new employee concentrates on his/her scientific background and work experience to provide the employee with a level of competence so that the individual will be able to function within the defined responsibilities of his/her position ASAP.

Training is a process used to assist laboratory personnel in their professional development. The training techniques utilized include:

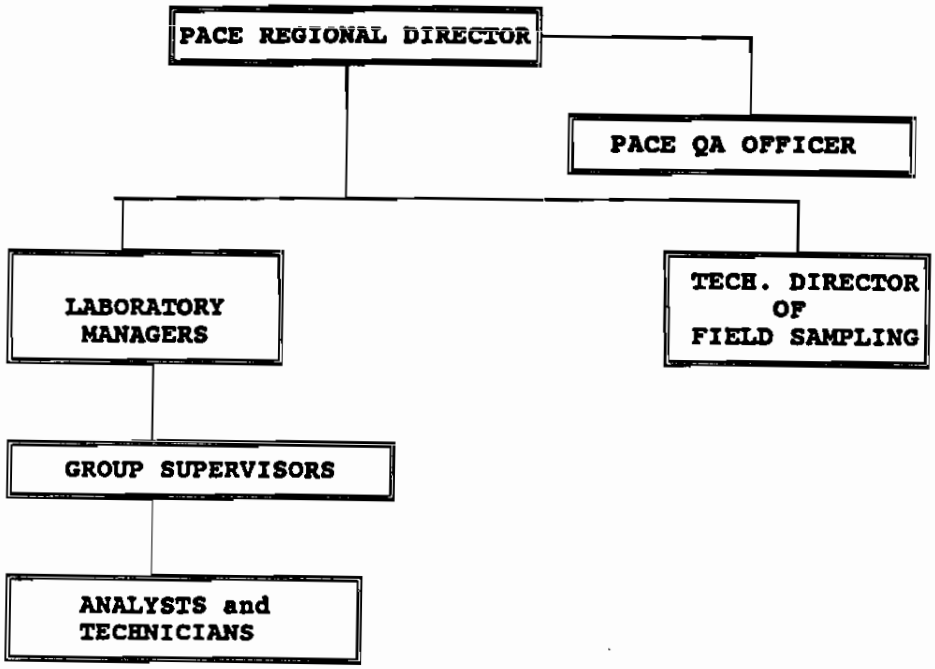
- on-the-job training
- lectures
- programmed learning
- conferences and seminars
- short courses
- specialized training by instrument manufacturers
- participation in check-sample or proficiency sample programs.

Group Supervisors shall be responsible for providing documentation of training and proficiency for each employee under their supervision. The Training Documentation File indicates what procedures (SOPs) a technician is capable of performing either solo or only with supervision. The files shall also include logbook pages and sample reports indicating performance of passing QC samples. The Group Supervisor is responsible for keeping training documentation updated and current. The QA department shall maintain a file for each technical employee. In addition to the training documentation, these files shall include a current CV and proofs of education such as college transcripts or certificates from continuing education coursework or specialized training.

Temporary employees receive the same orientation as permanent staff with the exception of the human resources orientation.

Figure 2-1

PACE New England, Inc.
QUALITY ASSURANCE
ORGANIZATION CHART



APPENDIX A

Resumes For PACE NE Technical Staff Employed As Of This Revision Date

DEBORAH F. MCGRATH
Regional Director
PACE New England, Inc.

Experience

As Regional Director, Ms. McGrath is responsible for the overall management of PACE New England which includes the oversight and coordination of Laboratory and Field Operations, Quality Assurance, Support Systems, Administrative, and Sales/Marketing departments. In addition, she serves as liaison for PACE NE on behalf of both clients and other laboratories within the PACE network.

Prior to joining PACE NE in 1993, Ms. McGrath served as Vice President and Division Manager of NET Inc., Cambridge Division, in Bedford, MA (formerly Cambridge Analytical Associates, Inc.), from 1989-1993 where her responsibilities included oversight of the technical operations and business development of the division. She was Vice President of Environmental Services for one year and Director of Analytical Services for two while employed by Cambridge Analytical Associates, Inc. from 1986-1989.

Ms. McGrath served in the position of Manager of the Laboratory Analysis Department and Inorganic Section while employed by GCA Technology Division, Bedford, MA from 1981-1986.

Previous to her management positions, Ms. McGrath held the position of analytical chemist at Kennecott Copper Ledgemont Laboratory, Lexington, MA.

Education

B.S. Chemistry, Simmons College, Boston, MA 1971

Publications

Ms. McGrath is the author/co-author of over 15 publications on methods development and comparison studies, waste oil and biota analysis, combustion assessments and waste minimization.

RICHARD L. WELLMAN
Operations Manager
PACE New England, Inc.

Experience

As Operations Manager, Mr. Wellman oversees management of both inorganics and organics laboratory groups providing wet chemistry, metals prep, metals, pesticide/PCB, volatiles and semivolatiles analysis. He reports to Ms. McGrath, Regional Director.

Mr. Wellman managed the Inorganic Laboratories for two years, previous to his present position. Prior to joining PACE, Mr. Wellman worked in similar capacities for Resource Analysts, Inc. which was acquired by PACE in June 1992. Mr. Wellman joined RAI in 1988 and worked as the GC Lab Director until 1991 when his responsibilities changed to managing the Metals Labs and the Reporting/Data Review and Lab Data Services Departments.

Mr. Wellman has extensive experience in the analytical chemistry field as a Consultant and Chemist. Mr. Wellman has over thirteen years of experience in Gas Chromatography, High Performance Liquid Chromatography, Infrared Spectroscopy, UV-VIS Spectrophotometry, GPC, and NMR.

Consultant, Medical & Scientific Enterprises, Sudbury, MA Summer 1986

Consultant, Acadia Management, Boston, MA Summer 1986

Systems Specialist, 1984-1985, Group Leader Analytical Chemistry, 1978-1984, DuPont/NEN Products, Boston, MA

Education

M.B.A., Business Administration, Boston University, Boston, MA, 1987

B.S., Chemistry, Union College, 1975

Continuing Education

"Environmental Data Requirements", conference, April 25, 1989

"HP RTE System Managers Course", May 22-26, 1989"

"Newest Analytical Methods and Techniques", Hewlett Packard Video Teleconference, April 19, 1990

STEPHANIE BECK
Quality Assurance Officer
Pace New England, Inc.

Experience

Ms. Beck is responsible for quality assurance activities at Pace New England, Inc. She is responsible for auditor training and Quality Assurance training for new employees. She performs internal inspections of PACE NE labs and is responsible for the implementation of quality programs at Pace NE, Inc. She reviews data for internal and regulatory compliance. Ms. Beck prepares Quality Assurance Project Plans. She reviews SOPs. Ms. Beck reports to Deborah McGrath, Regional Director.

Prior to joining PACE NE, INC. Ms. Beck worked in the same capacity for Resource Analysts, Inc. which was acquired by PACE NE, INC. in June 1992. Ms. Beck came to PACE NE, INC. with over 4 years laboratory experience performing inorganic chemistry analysis. Ms. Beck has over 2 years experience in the Quality Assurance department (1990 - present) performing the duties to assure management that facilities, equipment, personnel, methods, practices, records, and controls are adequate and in conformance with regulations. She has over 2 years experience reviewing Aquatic Tox. studies and Organic and Inorganic Chemistry data for FIFRA, TSCA, OECD, and FDA compliance as a GLP auditor. She has experience reviewing CLP data packages.

QA Officer, PACE NE, INC., Inc., Hampton, NH 5/92 - Pres

QA Auditor, Resource Analysts, Inc., Hampton, NH 4/90 - 5/92

Education

B.S., Environmental Economics, University of New Hampshire, 1988

Continuing Education

Environmental and Natural Resource Law, 1992 U. of N.H. Durham, NH
Introduction to Computer Validation SQA Annual Meeting 1991, Kansas City, MO
Intro. and Advanced WordPerfect 1991 OMC, Portsmouth NH
Producing Results with Others, 1991 Millipore, Bedford MA
SQA Basic Training GLP SQA Annual Meeting Oct 1990, Orlando FL
Lotus 123, 1990 Millipore, Bedford MA

THERESA I. HENNESSEY
Wet Chemistry Group Supervisor
New England Regional Office

Experience

Ms. Hennessey is responsible for managing and overseeing all Wet Lab workflow, analyses, spreadsheet review, raw data review, data handling and tracking, report generation, review and timely completeness. Ms. Hennessey supervises a staff of 7 - 8 technicians and is responsible for scheduling work hours, vacation and bi-annual performance and salary reviews. Ms. Hennessey reports to Mr. Wellman, Inorganics Laboratory Manager.

Prior to joining PACE, Ms. Hennessey worked in the same capacity for Resource Analysts, Inc. which was acquired by PACE in June 1992. While employed at RAI, Ms. Hennessey gained over six year's experience on the pH meter, Specific Conductance Meter, Zero Headspace Extractors, High Pressure Filtration System, Spectronic 20, Flashpoint Apparatus, Top Loading Balances, Analytical Balance and BTU. Ms. Hennessey has experience in the proper preparation of sample digests for trace metal analysis, including the digestion of tissues, solids, waxes, oils, plastics and polymers as well as the usual soils and water samples. Ms. Hennessey supervised the Metals Digests Lab for two and a half years with a staff of 3. For one year, she managed both the Wet Lab and Metals Digests Lab with a staff of 11-12. She has one year's experience in reviewing final Wet Lab data and reports and one year's experience in the use of Lotus Quattro.

Education

B.S., Environmental Conservation/Resource Economics, UNH 1984

Continuing Education

"General Chemistry" University of New Hampshire, Spring, 1987

"Leadership Skills for Women" March, 1988

"Introduction to Lotus", Millipore Corp., Bedford, MA, Nov. 1990

"Quantatative Analysis", University of New Hampshire 1992

"Organic Chemistry", University of New Hampshire 1992

CLIFFORD CHASE
Mass Spectrometry Supervisor
PACE New England, Inc.

Experience

Mr. Chase is responsible for supervision of the groups performing volatile and semivolatile GC/MS analyses as well as volatile GC analyses. Mr. Chase reports to Mr. Wellman, the Operations Manager.

Previous to his present position, Mr. Chase was responsible for supervision of the Metals Instrumentation laboratory for two years. Prior to joining PACE, Mr. Chase worked for Resource Analysts, Inc. which was acquired by PACE in June 1992. Mr. Chase joined RAI in 1988 and worked as an extractions analyst until 1990 when he became Organics Extractions Group Supervisor. Mr. Chase has over four years of supervisory experience and a strong background in instrumentation. He is experienced in the analysis of environmental samples and has 6 months training on the Extrel ELQ-400 GC/MS, the HP 5880 and HP 5840 GC/FID, and the HP 5890/5970 GC/MSD.

During a five year tenure at the University of New Hampshire, Mr. Chase was responsible for analyses, operator training, repair and maintenance of JEOL FX90Q FTNMR, Instrumentation Laboratory Model 951 AA and a Varian Cary 219 UV/VIS.

Education

B.S. Chemistry, University of New Hampshire, 1986

Continuing Education

"Mass Spectral Interpretation", University of New Hampshire, April/May 1988

"Aquarius GC/MS Data System Training Seminar", Hewlett Packard Co., Andover, MA, September 13-14, 1988

"Management Lead Skills Training", Millipore Corp., Bedford, MA, July 17, 24, 31, Aug. 7, 1990

"UV Detector Service Training", Waters Chromatography Division, MILLIPORE Corp., Marlborough, MA, April 20-21, 1992

I attest that the information in this Curriculum Vitae is true and accurate.

*Signature*_____ *Date*_____

PAUL RAITI
Organics Extractions Lab Supervisor
New England Regional Office

Experience

Mr. Raiti is responsible for the supervision of a staff of technicians performing sample preparation for ABN, PCB, PHC, PNA, pesticide and other organic methods. The Organics Prep Lab is also responsible for the oil and grease and phenol methods which Mr. Raiti oversees. As part of the organics sample preparation process, Mr. Raiti also performs and directs the operation of two GPC/HPLC units for sample clean-up. As supervisor, Mr. Raiti monitors all holding times and schedules daily workflow to ensure timeliness of all extract preparation. He is also responsible for the training of technicians to meet all PACE specific and general method requirements for data quality. As a supervisor-in-training, Mr. Raiti reports to Mr. Rhode, Organics Laboratory Manager, who has the ultimate responsibility for the Extractions Lab.

Prior to becoming the lab supervisor, Mr. Raiti had over two years experience as a Senior Technician in the Organics Prep Lab. He was hired as a Senior Tech by Resource Analysts, Inc. in 8/90 and continued to work in the same capacity after RAI was acquired by PACE in June 1992. Mr. Raiti is fully qualified in all organics sample preparation procedures performed at PACE.

Education

B.S., Plant Science, University of New Hampshire, 1989

A.S., Horticulture, Essex Agricultural and Technical Institute, 1987

Continuing Education

"Laws in Natural Resource and Environment", University of NH, Spring 1991

"Wetland Resource Management", University of NH, Fall 1991

"Analytical Chemistry", University of Massachusetts, 1992

PETER LEMAY
GC Operator/Group Supervisor
Radiation Safety Officer
New England Regional Office

Experience

Mr. Lemay is responsible for the analysis of PCB's, Pesticides, PNA's, PHC's, DI solvents, and Herbicides and for the supervision of three GC analysts. He reports to Mr. Rhode, Organics Laboratory Manager.

Prior to joining PACE, Mr. Lemay worked in the same capacity for Resource Analysts, Inc. which was acquired by PACE in June 1992. Mr. Lemay joined RAI in 1987. While employed at RAI, Mr. Lemay acquired three and a half year's experience on the following GC's: HP 5890, HP 5880, HP 5840, Tracon and waters dimension GC using the following detectors: ECD, ELCD, NPD, FID, FPD with either packed or capillary columns.

Mr. Lemay served as a chemist for Burgess Analytical Laborator, North Adams, MA from 1985 to 1987. His duties included performing chemical analyses on a variety of materials, sample preparation, operation of instruments, evaluating and reporting results, and training new employees.

Medical Laboratory Specialist, United States Air Force. 1977 - 1981

Education

B.A., Chemistry, University of Maine, Orono, ME 1985

Continuing Education

Intro to GC/MS - Hewlett Packard, 1988

Occupational and Environmental Radiation Protection, Harvard School of Public Health, Aug 1991

ELIZABETH A. DUPERE
Client Services Manager
PACE New England, Inc.

Experience	*Ms. Dupere is responsible for the operation of the Project Management and Sample Management Departments of PACE New England, Inc. Her duties include the management and supervision of the daily activities of these departments. Ms. Dupere reports to Ms. McGrath, Regional Director.* *Prior to her present position, Ms. Dupere supervised the Volatile Organics Department for one year. Before joining PACE NE, Inc., Ms. Dupere worked as an analyst for Resource Analysts, Inc. (RAI) which was acquired by PACE, Inc. in June 1992. She joined RAI in 1987 as a technician in the Wet Chemistry Lab. In 1988 she became a GC technician and, in 1990, a GC/MS technician. Ms. Dupere has more than three years experience in volatile organics analysis and two years experience in gas chromatography. She is experienced in the use of Hall, ECD, FID, FPD, and NPD detectors. As a Wet Chemistry lab technician, she received 10 months training in wet chemistry techniques.*
Education	*B.S., Biochemistry, University of New Hampshire, 1987*
Continuing Education	*"Use of Widebore Columns", By Restek, Inc., Boston, MA, December, 1989* *"Mass Spectral Interpretation", By Bill Rounds, University of New Hampshire, spring, 1990* *"Air Analysis Symposium" By Tekmar, Inc., Lexington, MA. May 1991.* *"GC/MS Troubleshooting", seminar, Hewlett Packard, Burlington, MA, Spring 1992*

CHRISTINE PAQUETTE STELZER
Senior Data Validator/Reporter
New England Regional Office

Experience

Ms. Stelzer is primarily responsible for the review, validation, and reporting of organics data as it pertains to special projects. Special projects include those under USEPA CLP protocol, state or government agency protocols, or unique PACE/client contracts. Part of her job involves reviewing Statements of Work, Quality Assurance Plans, and Bid Requests to identify special analytical, QC, or reporting requirements. Findings are communicated to Sales and Analytical staff to help ensure appropriate bidding and compliant analyses. Ms. Stelzer subsequently reviews organics data generated for these jobs to ensure accuracy and scientific defensibility. Ms. Stelzer is responsible for generating special forms and diskettes as required to complete the data package. She is the Project Manager/Data Reporting and Delivery Officer for the EPA CLP Organics Contract 68D20026. Ms. Stelzer reports to Mr. Rhode, Organics Laboratory Manager.

Prior to joining PACE, Mrs. Stelzer worked in the same capacity for Resource Analysts, Inc. which was acquired by PACE in June 1992. Ms. Stelzer joined RAI in 1987. She has worked as a Technician and Analyst in the Volatile Organics and Semivolatiles Lab. She has experience in GC/MS analysis using both packed and capillary columns under USEPA methods 624, 8240, 524.1, 501.3, 625, 8270 and 1988 CLP protocol. She is also experienced with gas chromatographs equipped with FID, PID, and Hall ELCD detectors for methods including 601, 8010, 602, 8020. Ms. Stelzer has thorough familiarity with the HP RTE software used for GC/MS data acquisition and reporting and is proficient in using HP LAS and LIMS data systems. She has a thorough understanding of USEPA CLP requirements for volatiles, semivolatiles, and pesticide/PCB's under 1988 and 1991 protocols.

Laboratory Technician, Environmental Testing Lab, Chicopee, MA. 1986 - 1987

Research Assistant, Elms College, Chicopee, MA. 1985 - 1986

Laboratory Assistant, Chemical Specialties, Springfield, MA. 1984

Education

B.A., Chemistry and Biology, Elms College, Chicopee, MA, Magna cum laude 1986

Continuing Education

"Systems Maintenance and Wastewater Treatment" Springfield Technical Community College, Springfield, MA, 1/19-5/29, 1987

Hewlett Packard RTE-6 Operator School by Hewlett Packard, New Orleans, LA, September 14-18, 1987

"Statistics" Springfield Tech. Comm. College, Springfield, MA, '87

"Custom Reporting", Hewlett Packard class by Hewlett Packard, 1988

Mass Spectral Interpretation Course University of New Hampshire, April 23 - May 18, 1988

Career Track Communication Skills Workshop August 17, 1988

Capillary Chromatography Seminar Restek, 12/11/89

"Train the Trainer", seminar by Maggie Dean, RAI, 4/92

Publications

Wright, M.L., and S.T. Jorey, Y.M. Myers, M.L. Fieldstad, C.M. Paquette, and M.B. Clark, 1988. Influence of Photoperiod, Daylength, and Feeding Schedule on Tadpole Growth and Development. Development Growth and Differentiation, 30, 315-323.

Wright, M.L. et al, 1988. Effect of Changing the Light/Dark Schedule, the Time of Onset of the Light or Dark Period, or the Daylength, on Rhythms of Epidermal Cell Proliferation. Chronobiology International, 5, 317-330.

Wright, M.L. and L.S. Blanchard, S.T. Jorey, C.A. Basso, Y.M. Myers and C.M. Paquette, 1990. Metamorphic Rate as a Function of the Light/Dark Cycle in Rana pipiens Larvae. Comp. Biochem. Physiol., 96A, 215-220.

KERRY HUNTER
Data Validator
New England Regional Office

Experience

Ms. Hunter is responsible for the review, validation and reporting of organics data (specifically pesticide/PCB packages) as it pertains to work conducted under specific protocols such as USEPA CLP protocol, state or government agency protocols or unique PACE/client contracts. Ms. Hunter is responsible for the generation of specific forms and diskette deliverables required to complete a data package. Ms. Hunter reports to Mr. Rhode, Organics Laboratory Manager and Technical Director.

Prior to her position as a Data Validator, Ms. Hunter worked in the Reporting Department where she was responsible for the generation of commercial reports for organic and inorganic analyses. Prior to joining PACE NE, Inc., Ms. Hunter worked in the same capacity for Resource Analysts, Inc. (RAI) which was acquired by PACE NE, Inc. in June 1992. Ms. Hunter joined RAI in 1986 and worked as a GC operator until 1990 when she became an analyst for the Product Registration Group (PRL) of RAI. In this capacity she was responsible for method validation and analysis of samples in support of GLP aquatic toxicology studies. In 1991, she was promoted to Senior Analyst for the PRL. Ms. Hunter has more than four years experience on the HP 5880, HP 5840, HP 5890 Capillary ECD, and the Tracor Hall, 5890 Hall, NPD, TCD, FID, LAS and EPA software, and HPLC experience using the Waters 490, 470, and 430 detectors.

Education

B.S., Biology, University of New Hampshire, 1985

Continuing Education

Laboratory Health and Safety - 1986

Fundamentals of LC - Hewlett Packard - 9/90

Radiation Safety Training course - Dr. George Chabot, Univ. of Lowell - 3/91

Waters service training for 420 and 470 detectors - Millipore Corp. - 4/91

STUART BRONSON
Senior Project Manager
PACE New England, Inc.

Experience	*Mr. Bronson serves as a Project Manager for clients who specialize in large scale, complex and on-going site work. He reviews Quality Assurance Project Plans for completeness, applicability and conformance to established protocols. QAPP information is condensed for Laboratory Project Alert Forms. Negotiates project specifications with clients, insures specifications are met by the lab and delivered to the client in the required time frame. Fields questions from a diverse clientele to determine testing needs; issues advice and answers technical questions about the lab, data, methodologies, protocols and procedures. He also maintains communication channels with the Sales Dept. and the Laboratory Staff and serves as the USEPA-CLP contact person for the lab. Mr. Bronson reports to Ms. Beth Dupere, Project Management/Sample Management Supervisor.* *Prior to joining PACE, Mr. Bronson worked as Director of Sample Management for Resource Analysts, Inc. which was acquired by PACE NE, Inc. in June 1992. Mr. Bronson joined RAI in 1990.* *Mr. Bronson worked at Laucks Testing Laboratory in Seattle, Washington for four years prior to RAI in the capacity of analyst and project manager.*
Education	*B.A. Ecology and Evolutionary Biology, University of Arizona, Tucson, Arizona, 1985.* *Coursework towards M.S. in Limnology, University of Washington, Seattle, Washington, 9/89 - 5/90.*
Continuing Education	*Customer Service Seminar. Dun and Bradstreet, held at Nashua, New Hampshire, May 1991*

GRETCHEN FRANZHEIM
Project Manager
PACE New England, Inc.

Experience

Ms. Franzheim currently serves as a Project Manager for clients specializing in large-scale, protocol-specific projects. She reviews Requests For Proposal (RFPs) and Quality Assurance Project Plans (QAPPs) for completeness, applicability and conformance to established protocols. She is responsible for communicating special project requirements to laboratory staff to ensure adherence to protocol or specific client project requirements. Ms. Franzheim serves as primary PACE NE contact with designated clients and is responsible for managing analytical projects from onset to completion. In addition, she fields questions from a diverse clientele to determine testing needs and to answer technical questions regarding protocol, methods, reporting and procedures. Ms. Franzheim reports to Ms. Dupere, Client Services Manager.

Previous to her position as a Project Manager, Ms. Franzheim functioned as a Senior Data Reviewer, Ms. Franzheim was responsible for the review of Inorganic CLP data and also served as a backup Organics CLP data reviewer. She is experienced in the review of environmental data for a wide variety of Organic and Inorganic Methods and Protocols including USEPA SAS (Special Analytical Services) projects, NJ ECRA, NYASP, NEESA, and HAZWRAP.

Prior to joining PACE, Ms. Franzheim worked for Resource Analysts, Inc. which was acquired by PACE in June 1992. She joined RAI in 1987 and served in the capacity of Data Review Supervisor until June 1992. Her responsibilities included the tracking of samples and documents through the analytical process while under contract to EPA. She supervised the assembly, review and validation of data for Organics CLP case packages, as well as handling the responsibility of training staff to perform these duties.

Ms. Franzheim has extensive experience in the chemistry field within a variety of areas. Ms. Franzheim gained experience in data review and quality assurance at Aquatec, Inc. and a Chemist at Ladd Research Industries, Inc. From 1977 to 1982, she taught chemistry, biology, physics, and mathematics in public schools.

Education

B.S., Chemistry, St. Lawrence University, 1975

M.S. Candidate, Environmental Studies, U. MASS, Lowell, MA

Continuing Education

"Seminar on CLP Organics", Technical Management Consulting, January, 1988

"Mass Spectral Interpretation", University of New Hampshire, Durham, NH, April - May, 1988

"In Search of Excellence", seminar by Tom Peters, October 18, 1988

"CLP/Superfund Automated Analytical Data Management Caucus", June 28-30, 1989

"Superfund Data Management Caucus", Ral[illegible] June 12-13, 1990

"Sixth Annual Waste Testing and Quality Assurance [illegible]posium", Washington, D.C., July 16-20, 1990

"Ward CLP Training Class", Ward Scientific, Ltd., Burlington, MA, Aug. 7-8, 1990

"Law of Natural Resources and Environment", Un[illegible]y of New Hampshire, Durham, NH, Spring 1991

"Industrial Pretreatment Pollution Prevention", by New England Interstate Environmental Training Center, Westford, MA, February 12, 1992.

"Environmental Law Update 1992", sponsored by Sh[illegible]n Phinney Bass + Green Professional Association, Nashu[illegible]. April 9, 1992.

"Environmental Chemistry Laboratory", University of Massachusetts, Lowell, MA, Fall 1992.

LILLIAN PEPIN
Project Manager
PACE New England, Inc.

Experience	*Ms. Pepin is responsible for project management from initiation to completion. Ms. Pepin is the point of client contact for projects, priorities, preliminary data, bottle orders and price quotes for commercial work and work by special protocol such as CLP. Ms. Pepin also serves as liaison for PACE Network projects in which PACE New England, Inc. is involved. Ms. Pepin reports to Ms. Dupere, Client Services Manager.* *Prior to joining PACE, Ms. Pepin worked in sample management for Resource Analysts, Inc. which was acquired by PACE in June 1992. Ms. Pepin joined RAI in 8/90 and worked in Sample Management where she was responsible for the receiving, handling, storage, and distribution of incoming samples. Her duties also included client contact, data entry, maintenance of sample storage area security, and sample separation for proper disposal.* *Ms. Pepin has significant previous experience in the health care industry and has pursued an education in data processing.*
Education	*Data Processing Certificate, Hesser College, Portsmouth, NH 1989* *Nursing Assistant Certificate, State of NH 1985*
Continuing Education	*"Exceptional Customer Service", Seminar, Dun & Bradstreet, Portland, ME May 1991*

DAVID BUCK
Computer Systems Manager
New England Regional Office

Experience

Mr. Buck is responsible for performing daily, weekly and monthly housekeeping duties on the PACE Network, LAS, and LABUX computer systems. He designs and implements new procedures for data entry and reporting. Mr. Buck is responsible for computer networks and their security. Mr. Buck reports to Mr. Jim McLeavey, Support Services Manager.

Prior to joining PACE, Mr. Buck worked in the same capacity for Resource Analysts, Inc. which was acquired by PACE in June 1992. Mr. Buck has had more than four years experience as a Computer Systems Manager and six years programming on Hewlett Packard 1000 computers, RTE-A/RTE-6/RTE-IVB. Mr. Buck has converted order tracking systems to data[illegible] systems and centralized collection of electronic test data.

6/92 - Pres Computer Systems Mgr., PACE, Inc., Hampton, NH

1988 - 6/92 Computer Systems Mgr. A.I. Hampton, NH

1987 - 1988 Computer Analyst, Sprague Electric, Sanford, ME

1985 - 1987 Computer Analyst, Fairchild Semiconductor, South Portland, ME

Education

M.B.A. Nasson College, Business Administration, 19[illegible]

B.S. University of Maine, Mathematics, 1982

Continuing Education

"Second Annual LIMS Conference", June, 1988

"LABSAM System Manager Course", January, 1989

"Novell System Manager", February 1991

"Unix Basics I and II", December 1991

"Unix System Manager", January 1992

3.0 OBJECTIVES

PACE NE is committed to the philosophy that quality operations result from quality planning, design, and work performance by skilled operational personnel. PACE NE's policy is to perform its varied types of technical work in accordance with standard quality assurance practices such as Good Laboratory Practices (GLP) and the EPA Contract Laboratory Program (CLP). PACE NE has a Quality Assurance Officer responsible for maintenance of standard operating procedures, laboratory audits, performance evaluations, federal and state certifications and quality assurance documentation.

Each laboratory worker is responsible for checking standard operating procedures when necessary; following these procedures during routine analyses; recording quality control information required by those procedures in the proper location, and taking appropriate corrective action including suspending analyses when quality control criteria are not met.

Objectives of the quality program are:

- to provide a quality organization independent of the pressures of project performance with the responsibility and authority for auditing and recommending corrective action;
- to provide a quality organization with clear paths of communication with management;
- to perform regularly scheduled audits and thereby document an objective evaluation of quality-related practices;
- to promptly identify variances and implement corrective actions;
- to maintain readily identifiable and retrievable records that provide documentary evidence of the quality of activities performed;
- to provide procedures for implementing project-specific quality plans;
- to define responsibility and authority for developing and implementing quality plans;
- to provide quality reference documentation for each project.

Quality Assurance objectives for measurement data can be expressed in terms of completeness, representativeness, accuracy, precision, comparability and traceability.

3.1 Completeness

Completeness is a measure of the amount of valid data obtained from a measurement system compared to the amount that was expected. The QA objective for completeness is to maximize the number of valid results. This can be attained by:

- minimizing sample loss and breakage
- performing sufficient QC samples to document control
- documenting all aspects of the analytical system

3.2 Representativeness

Representativeness is the extent to which reported analytical results truly depict the chemistry of the sampled environment. Representativeness is a qualitative objective which is optimized through proper selection of holding times and procedures, through proper sample preservation, and through prompt extraction and analysis. Refer to Table 6.1.

USEPA guidance is followed for sample preservation and field preservation is checked upon sample receipt in the laboratory. Refer to PACE NE SOP QA-400, Sample Receiving and Identification, current revision.

Sample holding times follow EPA recommendations. In cases where no formal recommendation has been made, the holding time for that analyte in a different matrix or a similar analyte in a similar matrix is applied.

3.3 Accuracy and Precision

Accuracy and precision data are optimized through the use of analytical procedures that minimize biases through the use of standard procedures, through the meticulous calibration of analytical equipment and by implementing corrective action whenever measured accuracy and precision exceed pre-established limits.

Accuracy and precision are assessed through the analysis of Laboratory Control Check Samples. Laboratory generated QC samples, such as method blanks and Laboratory Control Spike/Spike Duplicate Samples are used to assess the accuracy and precision of measurements due to laboratory activities. QC Samples, such as surrogate spikes and

matrix spike/matrix spike duplicates are used to monitor the effects of the sample matrix on precision and accuracy. QC Check Samples such as field blanks, field duplicates and trip blanks are used to assess the accuracy and precision of both sampling and laboratory activities. Accuracy and precision goals for the laboratory are based on laboratory historical data, specific method requirements and the requirements of each specific project. A more detailed discussion of these goals is provided in Section 11.

3.4 Comparability

Comparability is the extent to which comparisons among different measurements of the same quantity or quality will yield valid conclusions. Comparability is a qualitative objective that will be attained by utilizing standard techniques for sample analysis and by reporting analytical data in appropriate units. Comparability between PACE NE analytical results and those obtained by other researchers will be ensured through the use of EPA, ASTM, and other recognized methods.

3.5 Traceability

Traceability is the extent to which results can be substantiated by hard-copy documentation. Traceability documentation exists in two forms: that which links final numerical results to authoritative measurement standards, and that which explicitly describes the history of each sample from collection to analysis. Refer to Sections 6 and 10 for more specifics on PACE NE procedures.

4.0 STANDARD PRACTICES

4.1 Laboratory Safety

Sample receiving areas and laboratories shall be equipped with suitable hoods, respirators, protective clothing and eye wear, gloves, barrier creams and other measures to prevent or minimize staff contact with hazardous substances. Safety equipment such as eyewash stations, drench showers, spill adsorbents and neutralizers, fire extinguishers, first aid materials, and breathing oxygen shall be available.

As a matter of policy, PACE NE shall not accept known initiator explosives, known dioxin-contaminated materials or unusual biohazard materials. PACE NE shall accept only those radioactive materials for which the isotope and emissions are clearly identified, and are in compliance with the terms and conditions of any and all applicable license(s). PACE NE shall accept nitroaromatics and nitroamines providing that the client makes provisions for disposal of samples with a positive explosive identification.

A laboratory staff member shall be designated as Safety Manager by the Regional Director. The Safety Manager prepares and maintains safety-related SOP's, conducts safety and occupational health orientation, training and review sessions as required, and maintains up to date familiarity with safety and occupational health issues pertinent to the laboratory.

The Safety Manager prepares and maintains educational programs as required to comply with state and federal "right to know" legislation.

The Safety Manager or his designee shall conduct an orientation session with each new staff member to familiarize him/her with routine and emergency safety procedures and equipment. Generally, the first one to three days shall be devoted primarily to health and safety concerns. Eye protection and a lab coat shall be issued to the employee. A respirator will be issued, as required, after respiratory protection training. Refer to PACE NE NE SOP QA-607, Respiratory Protection. A tour of the laboratory shall be conducted. During the tour, needs for eye, skin, and respiratory protection shall be discussed as well as the use of safety glasses, face shields, goggles, partial and full-face respirators, ventilated work areas, fume hoods, gloves, barrier creams, and Tyvek coveralls. The location of eye wash stations, drench showers, fire extinguishers, and first aid equipment shall be shown to the employee and their use shall be described or demonstrated. Fire and spill notification, emergency procedures, and evacuation stations shall be taught during this session. Refer to PACE NE NE SOP QA-602, Employee Training Safety Tours. The orientation concludes with an introduction to potential

chemical hazards and the Material Safety Data Sheets (MSDS). MSDS shall be made available for review.
Employees shall be responsible for their own safety. Laboratory Manag Group Supervisors may require that certain levels of protective equipment be worn . n their judgement it is appropriate. Failure of an employee to wear required p.otective equipment will result in immediate disciplinary action.

4.2 Training

Laboratory Manager(s) shall be responsible for staff training programs, which may be administered by the Laboratory Managers and Group Supervisors.

Training shall be conducted for each individual on each procedure that he or she is to perform.

No individual shall conduct any analysis, experimental procedure or other professional function without continuous direct supervision until g in that procedure has been completed and the individual's ability to produce acc . been documented.

ining Documentation Forms and written records of training activities shall be generated and maintained by the Laboratory Managers or Group Supervisors.

Records of attendance at professional development seminars, conferences, ourses and the like shall be kept by the Quality Assurance Department in individual training files maintained for all technical staff. Refer to PACE NE SOP QA-556, Training Files.

4.3 Security and Confidentiality

Three tiers of security shall be maintained within all PACE NE, Inc. facilities with the purpose of controlling external influences on samples, analytical processes, and data. This helps assure the completeness, representativeness, accuracy, and precision of analytical results.

The first tier of security maintained shall be controlled access to laboratory buildings. Exterior doors to laboratory buildings shall remain either locked or continuously monitored by a PACE NE, Inc. staff member. Keyless door-lock combinations shall be changed every time an employee terminates employment at PACE NE. Posted signs shall direct visitors to the reception office and mark all other areas as off limits to unauthorized personnel. All visitors to the facilities must sign the Visitors' Logbook maintained by the receptionist. All visitors shall be accompanied b staff member during the duration of their stay on the premises. The staff memb shall escort the

visitor back to the reception area at the end of their visit where they shall sign out in the Visitor's Logbook. Prior to departure of the last staff member at the close of each day, all windows shall be locked and all doors checked and locked by the last staff member.

The second security level shall be within the facility and may be designated as required by the Laboratory Managers in consultation with the Regional Director. Individual Laboratory Managers or Group Supervisors may close specific areas under their responsibility to entry by unauthorized persons. A list of authorized persons shall be prepared and signed by the Regional Director. "Closed Areas" shall be designated by prominen postings at all points of access.

The final tier of security shall be comprised of specific secure areas for sample, data and client report storage which shall be lockable within the facilities, and to which access shall be limited to specific individuals or their designees. Security of sample storage areas shall be the responsibility of the Sample Manager. Refer to PACE NE SOP QA-401, Sample Storage. Security of samples and data during analysis and data reduction shall be the responsibility of Group Supervisors and Laboratory Managers. Security of client report archives shall be the responsibility of the Quality Assurance Officer. Refer to PACE NE SOP QA-550, Data Archives. These secure areas will be locked whenever these individuals or their designees are not present in the facility.

Designated laboratory sample storage locations are designed to limit access to authorized personnel only, and provisions for lock and key access shall be provided. No samples are to be removed without authorization, which consists of having a worklist requesting analysis on an aliquot and without filling out the associated chain-of-custody records.

Standard business practices of confidentiality shall apply to all documents and information regarding client analyses. Specific protocols for handling confidential documents are described in PACE NE SOP QA-557. Additional protocols for internal identification of samples and data by number only shall be implemented as required under contract-specific Quality Assurance Project Plans.

4.4 Traceability of Standards, Instrumentation, and Data

Since standard solutions used for analytical method calibration affect all data derived from the method, the importance of quality and traceability shall be paramount.

Laboratory staff may order only materials of certified purity from reputable suppliers. Records of reference material purchased shall be maintained by the appropriate department.

PACE NE strives to purchase only the highest quality materials. To that end, reference materials shall be NIST traceable, EPA certified (CRADA), or American Association for Laboratory Accreditation certified whenever possible.

If assayed materials are unavailable, the material of highest purity available shall be obtained and assayed in-house before use.

Reference material containers shall be identified with the standard serial reference number and dated upon receipt.

Bound laboratory notebooks shall be used by analysts and technicians to record preparation of working standards from identified reference material. The following information shall be recorded:

date of preparation
analyst's initials
source of reference material
amounts used
final volume
serial reference number of that stock solution

All standards containers shall be labelled with, at minimum (as on small glass ampules), the standard serial reference number; when possible, the name, concentration, date of preparation and expiration date of the stock standard.

All diluted working standards not consumed during an analytical session shall be labelled fully, including the serial reference number of any stock standard used in its preparation.

Instrumentation used shall be as prescribed in the SOP for the analytical method.

All instruments used to collect samples, generate sample results and/or reduce data shall be designated by a unique alphanumeric identifier. This instrument identifier shall appear on the instrument, in the analysts' notebooks, instrument logbooks and/or computer-generated hardcopy for all sample analyses.

Preventive maintenance shall be provided for all instruments and equipment as specified by the manufacturer, or as established by the Laboratory Manager (or Group Supervisor), whichever is more frequent. Preventive maintenance shall be conducted in order to assure timely, accurate and reproducible analytical processes in a safe laboratory or field environment.

All maintenance activities shall be recorded in either the instrument run log or a separate logbook unique to the instrument.

All data generated in and/or reported from the laboratory shall include reference to the person(s) who performed the analysis, the date of analysis, the method used, the identification of the instrument and the acceptability of the results in the context of the QC system.

The Laboratory Control Sheet issued to laboratory departments shall be used to initiate a laboratory data file for the project.

All data pertinent to sample preparation shall be recorded by the laboratory staff in bound notebooks with numbered pages, on preprinted bench sheet forms and/or in LABUX. If bench sheets are used, the original forms will be retained in a 3 ring binder and a true copy will be forwarded to the reporting group for inclusion with the archived report package. During the sample preparation process, a preparation sheet shall be prepared for the project by the preparation specialist. It shall contain the following information:

Sample identification numbers
Date of preparation
Method reference
Analyst's initials
Preparation weights and/or volumes
Relevant blank
Spike and surrogate data including the serial reference number and the instrumental analysis to be performed on each extract.

The preparation sheet shall be filed with the Laboratory Control Sheet in the Laboratory Project File.

At the time of sample analysis, the laboratory identification number, amount injected or otherwise analyzed, any dilution of the original sample and/or extract and other relevant sample data shall be entered into either an analyst's notebook or instrument logbook, or if possible into the instrument header.

All data relevant to the calculations should, where possible, be entered onto the instrument header including sample weight or volume, final volume, dilution, and spike level.

4.5 Accountability

All areas of the laboratory in which samples are received, stored, processed, or analyzed shall be kept in a condition that minimizes the risk of samples becoming lost or accidentally destroyed, contaminated, degraded, misidentified, improperly handled or otherwise compromised. The following practices shall be followed to assure that data reported represent results on the sample as submitted to the facility.

All employees shall be responsible for the cleanliness and order of their work areas. The Laboratory Manager(s) shall routinely tour the facilities noting major and minor infractions. These shall be brought to the attention of the respective group supervisors who formulate and institute corrective action through their staff.

Each sample, defined as a unit of matrix enclosed by a single container, shall be assigned a Laboratory Control Number by the Sample Management staff member who receives the sample. Provisions to identify field replicates and additional sample volume shall be incorporated into this procedure as described in PACE NE SOP QA-400, Sample Receiving and Identification.

Cross-referencing of Laboratory Control Numbers and Client Sample ID's shall be implemented in Sample Management documents as described in PACE NE SOP QA-400, Sample Receiving and Identification.

Sample analyses shall be identified by Laboratory Control Number in analysts' notebooks or instrument logbooks, which shall consist of bound laboratory notebooks with prenumbered pages or on pre-printed benchsheets.

Transfers of samples in and out of storage shall be recorded in Internal Custody Records maintained by the Sample Manager.

Standards shall be stored separately from samples and extracted samples.

Computerized systems for data generation designed in house shall be validated prior to implementation and shall contain provisions for password access and additional security measures as required.

Where commercial computer software is used for data generation within PACE NE, the software producer is responsible for validation of their system.

4.6 Sample Analysis

Samples shall be analyzed within holding times as specified in Table 6.1, unless a more restrictive holding time is prescribed under a contract-specific QA Project Plan.

Samples shall be analyzed and experimental procedures conducted following written Standard Operating Procedures (SOP's) which have been approved in writing by management. Where PACE NE SOPs differ from EPA or Standard Methods, these differences will be noted in the SOP. Refer to Section 8.0, Analytical Procedures. Substantial changes to established procedures shall be authorized in writing by management via an SOP revision process as described in PACE NE SOP QA-553, Preparation of SOP's.

Departures from SOP's need management approval prior to implementation and shall be recorded in the raw data and on an SOP Deviation Form obtained from the QA Department (Figure 12-1). A copy of the form should be filed with the associated report file.

Laboratory Managers shall assure that samples are scheduled for analysis in compliance with the analytical request as issued by the Sample Manager in the form of a Laboratory Control Sheet(s).

A written Worklist System approved by the Technical Director shall be used to assign work to preparatory and instrumentation lab staff members.

4.7 Data Validation

Each department shall have written procedures for data validation which incorporate the quality assurance goals of traceability, accountability completeness, precision and accuracy.

No written reports shall be issued which have not undergone the data validation process.

4.8 Documentation

All information related to the quality assurance practices outlined in this manual shall be contained in records. This shall include, but not be limited to, standard operating procedures, study protocols, results of instrument calibrations, analysis of quality control samples, analysis of samples, sample custody and disposal, preparation of standards, corrective action reports, audits and inspections.

The Quality Assurance Office shall keep written inventories of quality-related documents.

5.0 MATERIALS AND APPARATUS

5.1 Reagents, Solvents and Gases

Chemical reagents, solvents, gases, and standards, supplied by reputable chemical suppliers, are used in the laboratory. All chemical reagents used for analyses shall be at least "Analytical Reagent Grade". Individual method references may indicate specific reagent requirements.

Materials are dated upon receipt in the laboratory. Solvents are checked for purity before use (Section 5.2.2, Solvent Lot Checks). Filters are placed on gas lines supplying instruments as an extra precaution.

All solvents and gases used shall be chosen to assure compliance with specific method and SOP requirements.

5.2 Laboratory Equipment

5.2.1 Refrigerator/Freezer Temperature Logs

Refrigerators and freezers are checked every weekday to ensure that they are operating properly and within established temperature ranges. All information is recorded on monthly data sheets taped to the door of each unit. Routine maintenance such as defrosting is performed as needed. Refer to PACE NE SOPs QA-800 and QA-801. Responsibility for performing the checks is assigned within the laboratory section where the units are located. The QA Department is responsible for ascertaining that checks have been performed and that necessary corrective actions have been instituted when needed. The QA Department is responsible for maintaining all historical temperature logsheets.

5.2.2 Solvent Lot Checks

Solvents are checked for trace contaminants on a lot-by-lot basis. When a new lot is opened, the chromatogram for the blank associated with the first samples extracted using this new lot is checked for any contamination. All information relevant to the check is recorded and maintained in a Solvent Check File. Responsibility for performing the checks and maintaining the records is assigned to the Extractions Laboratory Group Supervisor.

5.3 Glassware

All glassware used in the laboratory is maintained in good condition, cleaned, properly stored, and separated according to its specific laboratory application. Cracked, excessively chipped or otherwise defective glassware is either discarded or repaired. PACE NE's analytical laboratory purchases the majority of glassware from recognized commercial laboratory glassware suppliers such as Fisher Scientific and Baxter. All volumetric glassware utilized is class "A" certified.

Each laboratory maintains its own set of glassware, completely independent from the other laboratories. Cleaning of glassware is performed in each preparation laboratory to ensure that the glassware remains within each laboratory.

5.4 Glassware Cleaning

Laboratory glassware is scrupulously cleaned prior to use. Different cleaning procedures exist for different types of analyses and glassware. Refer to PACE NE SOP QA-802 (inorganics), QA-805 (metals preparation), and QA-804 (general organics).

5.5 Sample Preservation and Storage

Samples are preserved according to the EPA's recommendations (refer to Table 6.1) unless otherwise instructed, and stored to minimize sample contamination. To keep samples of differing levels of contamination separate, samples are segregated when high levels of contamination are known to be present. The laboratory must rely upon information supplied by the sampling team to document any known hazards. If there are samples that are suspected of having contaminants at high levels, they are unpacked in a hood. For Log-In procedures and supporting documentation, see Section 6 and PACE NE SOPs QA-400 and QA-401.

A holding blank comprised of DI water is placed into the volatiles storage refrigerators in the sample Receiving Area to monitor the potential of cross contamination. The holding blank is created daily for volatiles and analyzed whenever the associated jobs are analyzed.

5.6 Instruments

Laboratory instrumentation used shall be as specified in the protocol for the analytical method. Table 5-1 is a listing of major analytical instrumentation present in this laboratory.

Preventive maintenance is performed for each instrument by analysts and technicians on an ongoing basis and the activities documented in a bound instrument maintenance logbook or in the analysts runlogs.

Corrective maintenance shall be provided as required for all instruments and equipment and documented in appropriate logbooks. Factory replacement parts, trained service technicians and first quality materials shall be used whenever available. It is PACE NE's policy to conduct repairs at the lowest level of complexity necessary and to obtain parts directly from primary manufacturers whenever possible. The purpose of this policy is to maintain efficiency, economy and reliability of quality maintenance.

TABLE 5-1

ANALYTICAL INSTRUMENTATION

ITEM	QTY.	DESCRIPTION
GC/MS & GC SYSTEMS:		
GC/MS/DS	2	HP 5970 on RTE A900 HP-1000
GC/MS/DS	2	HP 5970 on RTE A900 HP-1000
GC/MS/DS	2	HP 5970 on RTE 600 HP-1000
GC/MS/DS	1	Extrel ELQ 400 on Digital PDP-11
GC/(2ECD)	2	HP 5890
GC(2ECD)	1	HP 5880
GC(FID, FPD)	1	HP 5890
GC(NPD, ECD)	1	HP 5890
GC(FID, ECD)	2	HP 5840, HP 5890
GC(FID, TCD)	1	HP 5880 dual channel
GC(PID, HECD)	1	Tracor 540
GC(HECD,ELCD)	2	HP 5890
GC(FID)	1	HP 5890
GC(NPD-FID)	1	Waters Dimension 1
GC(Hall-PID)	1	Waters Dimension 1
Auto Sampler	9	HP 7673A
Auto Sampler	1	Tekmar ALS 2016
Auto Sampler	5	Tekmar ALS
Purge & Trap	2	Tekmar LSC 2000
Purge & Trap	5	Tekmar LSC 2
Dynamic Headspace Concentrator	1	Tekmar 4000/4200
Thermal Desorber	1	Century ATD
Auto Sampler	2	HP 7672A
Auto Sampler	1	Varian 8050
Auto Sampler	2	Dynatech PTA-30
HPLC SYSTEMS:		
LC System-Auto Sampler	6	Waters Assoc. 48 position 712 WISP
LC System-Chromatography Pumps	5	Waters Assoc. 510
LC System-Conductivity Detector	1	Waters Assoc. 431
LC System-Fluorescence Detector	1	Waters Assoc. 420
LC System-Pump Controller	3	Waters Assoc. 680
LC System-RI Detector	1	Waters Assoc. 410
LC System-UV Spectrophotometer	2	Waters Assoc. 490E
LC System-UV Spectrophotometer	1	Waters Assoc. 441
LC System-Chromatography Pump	1	Waters Assoc. 590

TABLE 5-1 - CONTINUED

ANALYTICAL INSTRUMENTATION

ITEM	QTY.	DESCRIPTION
HPLC SYSTEMS - CONTINUED:		
LC System-Fraction Collector	1	ISCO - "Foxy"
LC System-Fraction Collector	1	Waters Assoc.
Post Column Derivatization System	1	Waters Assoc.
LC System-WAVS	1	Waters Assoc. Auto. Valve Station
Ion Chromatograph	1	Dionex 2000i
LC System-UV Detector	1	LDC Spectromonitor II
LC System-Scanning Fluorescence Detector	2	Waters Assoc. 470
LC System-Tunable Absorbance Detector	2	Waters Assoc. 484
LC System Controller & Pump	1	Waters Assoc. 600E
LC System Temperature Control Module	1	Waters Assoc. TCM
METALS INSTRUMENTATION:		
ICP	1	Thermo Jarrell Ash ICAP 61
ICP	1	Leeman Labs Plasma Spectrophotometer
AA Spectrophotometer	1	Hitachi Z9000 Zeeman (4 channel)
AA Spectrophotometer	1	Varian Spectra AA 20AA
AA Spectrophotometer	1	Perkin-Elmer 5100 Zeeman Furnace
AA Spectrophotometer	1	Perkin-Elmer 3100
Vapor Generation Accessory	1	Varian VGA-76
Auto Sampler	1	Varian PS-56
Auto Sampler	1	Perkin-Elmer AS90
Auto Sampler	1	Perkin-Elmer AS60/70
Furnace Atomizer	1	Perkin Elmer HGA 600
Mercury/Hydride System	1	Perkin-Elmer FIAS 200
DATA MANAGEMENT EQUIPMENT:		
Lab Automation System (LAS)	1	HP 3350 on RTE A900 HP-1000
Lab Information System (LABSAM)	1	HP on RTE A900 HP-1000
Tape Backup System (LABSAM, LAS)	1	HP 35401
Tape Data Archival (GC/MS)	2	HP 7970B
Tape Data Archival (GC/MS)	2	HP 7974
LAS/LABSAM Terminals	22	HP B&W/Color/150 Terminals
Personal Computers	85	NEC, CompuAdd & Compaq
Organic CLP Software	-	Finnigan Formaster
Inorganic CLP Software	-	Telecations

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TABLE 5-1 - CONTINUED

ANALYTICAL INSTRUMENTATION

ITEM	QTY.	DESCRIPTION
OTHER INSTRUMENTATION:		
Autoanalyzer	1	Lachat QuikChem AE
IR Spectrophotometer	1	Perkin Elr [illegible]420
Total Organic Carbon Analyzer	1	Astro 20[illegible]
Total Organic Halide Analyzer	1	Dohrman
Liquid Scintillation Counter	1	LKB [illegible]e 1214 Rackbeta
UV-Vis Spectrophotometer	1	Bau[illegible] & Lomb Spectronic 601
Visable Spectrophotometer	2	Milton Roy 301
Analytical Balance	2	Mettler AE200
Analytical Balance	1	Mettler H31AR
Analytical Balance	1	American Sci. Prod. SP182
Top-loading Balance	1	[illegible]erican Sci. Prod. Z3000
Top-loading Balance	1	[illegible]rican Sci. Prod. Z410
Top-Loading Balance	1	[illegible]er PJ400
Top-Loading Balance	1	[illegible] EK-1200A
pH Meter	1	[illegible]an 43
pH Meter	1	[illegible]an 11
pH Meter	1	[illegible] Electronics 6071
Conductivity Meter	1	[illegible]Scientific Model 32
Rotary Extractors	4	[illegible]ciated Design Model 3740-12-BRE

6.0 SAMPLE CUSTODY

Chain-of-Custody encompasses three major elements: field sampling, laboratory analysis and final data file. A Chain-of-Custody (COC) document may be the means in some types of legal proceedings by which evidence of custody of samples from time of receipt to completion of analysis is proved in the courts. PACE NE has implemented standard operating procedures to ensure that sample custody objectives of traceability and responsibility are achieved for every project. This section covers quality related activities from the receipt of samples at the laboratory through the issuance of final analytical data and the storage of data in its final data file.

6.1 Chain-of-Custody

The National Enforcement Investigations Center (NEIC) of EPA defines evidence of custody in the following manner:

1. It is in your actual possession, or
2. It is in your view, after being in your physical possession, or
3. It was in your possession and then you locked or sealed it up to prevent tampering, or
4. It is in a secure area.

Samples may be physical evidence and should be handled according to certain procedural safeguards. Field personnel or Client representatives complete a Chain-of-Custody Form for all samples. Samples are received by the laboratory accompanied by these forms.

The sampler should provide the following information:

- Client project name
- Project location
- Field sample number/identification
- Date and time sampled
- Sample type

- Preservative
- Analysis requested
- Sampler signature
- Signature of person relinquishing samples
- Date and time relinquished
- Sampler remarks
- Custody Seal Number

The record is filled out completely and legibly. Errors are corrected by drawing a single line through and initialing and dating the error. The error is also "error coded" to explain the reason for the correction (refer to Table 9-1). The correct information is then recorded with indelible ink. All transfers of samples except to and from commercial couriers must be recorded on the Chain-of-Custody via the "relinquished" a "received by" sections. All information except signatures may be printed.

When samples are received into the laboratory (refer to Section 6.3), Sample Management Personnel sign the Chain-of-Custody, verify their integrity as they are unpacked and explicitly state in the receipt records whether the Chain-of-Custody seal is intact, whether the sample is received intact or broken, and whether the sample is appropriately identified. This information is documented the Sample Receipt Condition Report (SRCR) and the client is contacted if any discrepancies or problems are found. If the integrity requirements are met or when any discrepancies are solved, the sample is assigned a laboratory identification number, the sample is stored in the appropriate refrigerator and the pertinent information is entered into the Laboratory Information Management System (LIMS). Once samples are in the laboratory, an Internal Custody Record is generated to track the transport and status of each sample within the laboratory. After sample log-in, a project file is started by the Sample Management Group Supervisor which includes the Chain-of-Custody record and all sample receipt documentation. Further detail on internal laboratory custody procedures is provided in subsequent subsections.

6.2 Sampling Kits

In general, sampling kits comprise the following:

- Sampling containers
- Preservatives (upon request) and appropriate MSDSs
- Chain-of-Custody forms
- Custody Seals (upon request)
- Sample labels
- Packing Material
- Shipping containers
- Ice Packs

Sample kit requests are received by the laboratory personnel (generally the Sample Manager) from a Project Manager or sampling team members via telephone request, memo, or facsimile.

Based upon the specific request, the Sample Manager or Project Manager determines the appropriate containers, preservatives and the necessary volume/quantity to specify for the analysis. This information is outlined in Tables 6-1. Refer to PACE NE SOP QA-402, Bottle Orders and QA-403.

6.2.1 Sample Containers

VOA vials (40 mL) are purchased pre-cleaned from Industrial Glassware.

Sample bottles for all analyses other than VOA are purchased from Fisher Scientific. All glass and plasticware is purchased in lots of about 15-50 cases per size and type of bottle.

An internal lot number is assigned to each batch of bottles purchased so that each batch may be tracked and tested for cleanliness. One or two bottles per batch per type of analysis is taken at random and tested. PACE NE tests its glassware for cyanide, 23 metals, acid/base/neutrals, pesticides, PCBs, and volatile organics as follows:

Table 6-1

Sampling and Preservation Requirements

Parameter	Method	Required Volume & Containers[3]	Preservation	Holding Time (in days)
Organics:				
Drinking water GC/MS VOA	EPA 524.1/524.2	3 x 40 ml Glass vial	HCl; 4°C[2]; No headspace	14
Wastewater GC/MS VOA	EPA 624/SW846 8240	2 x 40 ml Glass vial	HCl; 4°C[2]; No headspace	14
Purgeable Halocarbons	EPA 601/SW846 8010	2 x 40 ml Glass vial	HCl; 4°C[2]; No headspace	14
Aromatics	EPA 602/SW846 8020	2 x 40 ml Glass vial	HCl; 4°C[2]; No headspace	14
Acrolein, Acrylonitrile	EPA 603/SW846 8030	2 x 40 ml Glass vial	pH 4-5; 4°C[2] No headspace	14
TOX (Total Organic Halides)	subcontracted	250 ml Glass,amber	H_2SO_4; 4°C; No headspace	7
Acids/Base Neutrals	EPA 625/SW846 8270	1000 ml/30 g Glass,amber	4°C[2]	7(14)/40
Pesticides/PCB's	EPA 608/SW846 8080	1000 ml/30 g Glass	NaOH,H_2SO_4,pH 5-9;4°C[2]	7(14)/40
Carbamate Pesticides	EPA 531.1	120 ml Glass	3.6 ml Monochloroacetic acid buffer pH 3; Freeze	28
Herbicides (SDWA)	SM 509A/SW846 8150	1000 ml/30 g Glass	4°C[2]	7(14)/40
Petroleum Hydrocarbons-GC/FID	ASTM D-3328-78	1000 ml/30 g Glass	H_2SO_4; 4°C	7(14)/40[6]
Petroleum Hydrocarbons-IR	EPA 418.1	1000 ml/30 g Glass	H_2SO_4; 4°C	28
Polynuclear Aromatics	EPA 610/SW846 8100	1000 ml/30 g Glass,amber	4°C[2]	7(14)/40
Oil & Grease - Gravimetry	EPA 413.1	1000 ml/30 g Glass	1+1 HCl; 4°C	28
Oil & Grease - IR	EPA 413.2	1000 ml/30 g Glass	1+1 HCl; 4°C	28
NM2P	EPA 625/SW846 8270	1000 ml/30 g Glass	4°C	7(14)/40
Total Phenolics	EPA 420.3	200 ml/30 g Glass,amber	H_2SO_4; 4°C	28
Dioxins	subcontracted	1000 ml/100 g Glass	4°C[2]	7(14)/40
Metals:				
Mercury	245.1/7470 (7471)	50 ml/10 g	HNO_3	28
Arsenic & Selenium	EPA 200/7000 Series	50 ml/10 g	HNO_3	180
Lead, Thallium, Antimony	EPA 200/7000 Series	50 ml/10 g	HNO_3	180
All other metals (ICP metals)	EPA 200.7/SW 6010	50 ml/10 g	HNO_3	180
plus Hardness	SM 314A/EPA 200.7	(Included)	HNO_3	180

The following elements can be analyzed by ICP: Al, Ag, B, Ba, Be, Ca, Cd, Co, Cr, Cu, Fe, K, Mg, Mo, Mn, Si, Sr, Sn, Na, Ni, Pb, Ti, V and Zn in water, wastewater, and digested solids. As, Pb (water), Se, Tl and other metals normally analyzed by furnace may be determined by ICP but only with reduced sensitivity and therefore higher detection limits. Hg is analyzed by cold vapor.

NOTE: If duplicates and spikes are required, triple the sample volume for water samples.

EPTOX	SW846 1310	250 grams	N/A	N/A
TCLP	Fed.Reg.Vol. 55 June 29, 1990 Rules and Reg.	250 grams (2 X 250 g containers if VOAs are required)	4°C	See Below

	FROM COLLECTION TO TCLP EXTRACTION	FROM TCLP EXTRACTION TO PREPARATIVE EXT'N	FROM PREP EXT'N TO ANALYSIS
VOLATILES	14	N/A	14
SEMIVOLATILES	14	7	40
MERCURY	28	N/A	28
METALS EXCEPT MERCURY	180	N/A	180
PESTs/HERBs	14	7	40

Table 6-1, continued

Sampling and Preservation Requirements

Parameter	Method	Required Volume & Containers[3]	Preservation[7]	Holding Time (in days)
Inorganics:				
Acidity	EPA 305.1	125 ml/ 2 g	4°C	14
Alkalinity	EPA 310.1	125 ml/ 2 g	4°C	14
Asbestos	subcontracted	1000 ml Glass	4°C	
Bromide	EPA 300.0	10 ml/20 g	4°C	28
BOD	EPA 405.1	250 ml	4°C	2
BTU	ASTM D 240	5 ml/ 2 g	4°C	N/A
Chloride	EPA 300.0 or 325.1	10 ml/20 g	4°C	28
Chlorine upon Combustion (oil)	ASTM D 129-64/300.0	20 g	4°C	N/A
Chlorine, Residual	APHA 4500-CLG	200 ml		Field Test
COD	EPA 410.4	50 ml/ 5 g	H_2SO_4; 4°C	28
Coliform Bacteria	subcontracted	125 ml sterile	4°C[2]	24(6) Hours[5]
Color	EPA 110.2	100 ml	4°C	2
Conductance	EPA 120.1	100 ml	4°C	28
Cyanide, Total	EPA 335.2/SW 9010	1000 ml/25 g	NaOH; 4°C[1]	14
Cyanide, Ammenable	EPA 335.1/SW 9010	1000 ml/25 g	NaOH; 4°C[1]	14
Flashpoint	ASTM D 93-77	80 ml/80 g	4°C	N/A
Fluoride	EPA 340.1/340.2	100 ml (NPDES=500ml) Plastic	4°C	28
Formaldehyde	NIOSH 3500 mod	20 ml/20 g	4°C	N/A
Grain size (dry sieving)	EPA/CE-81-1	100 g		N/A
Hexavalent Chromium	SW846 7196	125 ml/20 g	4°C	1
Ignitability	SW846 1010	50 ml/25 g	4°C	N/A
MBAS (Surfactants)	EPA 425.1	150 ml	4°C	2
Nitrogen - Ammonia	EPA 350.1 or .3	200 ml/ 5 g	H_2SO_4; 4°C	28
" - Nitrate	EPA 300.0 or 353.2	25 ml/20 g	4°C	2
" - Nitrite	EPA 300.0 or 354.1	25 ml/20 g	4°C	2
Nitrogen - Nitrate+nitrite	EPA 353.2	25 ml/20 g	H_2SO_4; 4°C	28
" - TKN	EPA 351.3	200 ml/ 5 g	H_2SO_4; 4°C	28
" - TON (NH_4 & TKN)	EPA 350.3/351.3	250 ml/ 5 g	H_2SO_4; 4°C	28
Ortho Phosphate-P	EPA 365.1 or .3	100 ml	4°C	2
Particle size (wet digestion)	EPA/CE-81-1	100 g	4°C	N/A
pH	EPA 150.1	25 ml		Field Test
Phosphorus, Total	EPA 365.3 or .4	100 ml/ 5 g	H_2SO_4; 4°C	28
Radiology - R, U, Alpha,Beta,Gamma	subcontracted	3000 ml	HNO_3; 4°C	180
Radon	subcontracted	2 x 40 ml Glass	4°C	2
Reactivity- Sulfide Spot Test	SM 427.3C	10 ml/10 g	4°C	N/A
Reactivity- Cyanide Spot Test	SM 412J	10 ml/10 g	4°C	N/A
Reactivity- Releasable Cyanide	SW 846,Sec.7.3.3.2	10 g	No Headspace, 4°C	N/A
Reactivity- Releasable Sulfide	SW 846,Sec.7.3.4.1	10 g	No Headspace, 4°C	N/A
Silicate, Reactive	EPA 370.1	100 ml/ 2 g Plastic	4°C	28
Solids, Settleable	EPA 160.5	1000 ml	4°C	2
Solids, Total (TS)	EPA 160.3	100 ml[4]	4°C	7
Solids, Total Suspended (TSS)	EPA 160.2	100 ml[4]	4°C	7
Solids, Total Dissolved (TDS)	EPA 160.1	100 ml[4]	4°C	7
Solids, Total Volatile (TVS)	EPA 160.4	100 ml[4]	4°C	7
Solids, Total Suspended Volatile	EPA 160.4 or .2	100 ml[4]	4°C	7
% Solids (% moisture)	APHA 2540G	30 g	4°C	N/A
Specific Gravity	EPA/CE 1981	30 ml	4°C	N/A
Sulfate	EPA 300.0 or 375.2	10 ml/20 g	4°C	28
Sulfide	EPA 376.2	250 ml	ZnAc;NAOH,pH>9; 4°C	7
Sulfite	EPA 377.1	100 ml		Field Test
Tannins and Lignins	APHA 5550B	100 ml	4°C	N/A

Table 6-1, continued

Sampling and Preservation Requirements

Parameter	Method	Required Volume & Containers[3]	Preservation[7]	Holding Time (in days)
Inorganics:				
TIC (Total Inorganic Carbon)	EPA 415.1 mod	60 ml/ 2 g	4°C	28
TOC	EPA 415.1	60 ml/ 2 g	No Headspace H_2SO_4; 4°C	28
Turbidity	EPA 180.1	100 ml	4°C	2
Viscosity	ASTM D-445	300 ml	4°C	N/A

References:

EPA = 40 CFR 136
SW846 = Test Methods for Evaluating Solid Waste Physical/Chemical Methods, US EPA SW846, 3rd edition, 1986
SM = Standard Methods For The Examination of Water and Wastewater, 16th ed., 1985
APHA = Standard Methods For The Examination of Water and Wastewater, 17th ed., 1989
ASTM = American Society for Testing and Materials

[1] .006% ascorbic acid if residual chlorine is present.
[2] .008% sodium thiosulfate if residual chlorine is present.
[3] Plastic or glass jars are suitable unless otherwise indicated.
[4] TSS, TDS and TSVS are performed on the same 100 ml sample. TS & TVS are performed on the same 100 ml sample.
[5] "Holding time is not to exceed six hours if the data is to be used in litigation."
[6] No holding time is published - these times are suggested/borrowed from the ABN hold time.
[7] Acid preservations (H_2SO_4, HNO_3, HCl) are adjusted to pH < 2, base preservations (NaOH) are adjusted to pH > 12 unless otherwise noted.

Cyanide: Fill a one liter plastic bottle with DI water and preserve with NaOH. Analyze the water according to normal laboratory procedures.

Metals: Fill a 500 mL poly bottle with 100 mL of 2% nitric acid solution from the metals instrumentation room. Shake. Analyze for metals.

ABNs, Pesticides, PCBs: Submit an empty 1 L glass jar to the Organics Extractions Laboratory for testing with instructions for special extraction procedures to be employed as follows - Add 60 mL $MeCL_2$, shake for 2 minutes, KD to appropriate test volume and analyze under normal laboratory procedures.

VOA: Submit an empty 120 mL jar to the volatiles laboratory with instructions to fill the jar with VOA-free DI water. Let stand for 24 hours and analyze under normal laboratory procedures.

If any analyses indicate contamination, the affected lot is washed according to one of the following protocols and a random sample is retested.

Cleaning Procedure 1 (Extractable Organics)

- wash glass bottles, teflon* liner and caps in hot tap water with laboratory grade non-phosphate detergent
- rinse with tap water
- rinse with 10% HCl (metals-grade HCl in ASTM deionized water)
- rinse three times with ASTM type 1 deionized water
- rinse with hexane
- rinse with pesticide grade methylene chloride
- air dry or oven dry at 103°C
- place liners in lids and cap containers

Procedure 1 is used for amber and clear glass wide mouth jars, amber and clear glass Boston round, and amber and clear glass jugs.

Cleaning Procedure 2 (Purgeable Volatile Organics)

- bake opened jars and lids in a 103°C oven for 30 minutes
- cool
- cap

Procedure 2 is used for 40 ml glass vials, amber and clear, open and closed top, and 8 oz. amber Boston round with septa top.

Cleaning Procedure 3 (Metals, Cyanide)

- wash polyethylene bottles and caps in hot tap water with laboratory grade non-phosphate detergent
- rinse with tap water
- rinse with 10% HCl (metals-grade HCl in ASTM deionized water)
- rinse with deionized water
- invert and air dry in contaminant-free environment
- cap bottle

Procedure 3 is used for HDPE modern round bottles and cubitainers.

6.2.2 Assembling Kits

All glass containers are to be surrounded with packing material to prevent damage.

All preservatives are stored in 10 - 20 ml plastic squeeze bottles, contained within 250 ml plastic wide mouth jars which are appropriately labelled.

The appropriate number of labels and COC's are affixed to the ziplock plastic bag that the sample is stored in.

All the above contents are to be placed inside a cooler with ice packs (if requested) and secured for shipment using styrofoam (packing peanuts or other appropriate packing materials).

Sample kits are delivered to the sampling team via Federal Express, UPS, courier, or personally picked up by the sampling team.

6.3 Sample Receipt and Log-In

Refer to PACE NE SOP QA-400, Sample Receiving and Identification.

Typically, samples are received by the laboratory during normal business hours (8:00 am to 6:00 pm), Monday through Friday and 8:00 am to noon on Saturday.

Shipments for after hours and Sunday delivery are prearranged with laboratory personnel to ensure that personnel will be available to sign the airbill, record the date and time of sample receipt and to place the cooler in the sample management area under refrigeration until the next business day.

Upon sample receipt, the coolers are inspected for the general condition of the Custody Seal, if present. The coolers are then opened and each sample is inspected for damage. The sample containers are removed from the packing material and identities are verified against the Chain-of-Custody. All information regarding sample condition upon receipt is documented on the Sample Receipt Condition Report (SRCR). The report documents:

- Name of person if hand delivered
- Presence/Absence of COC forms and custody seals
- Condition of the custody seals, if present
- Discrepancies noted
- Holding times and preservatives
- Proper sample containers
- Appropriate sample volume

The Sample Receipt Condition Report is completed by signing and recording the date and time. If there are any discrepancies or problems with the samples or documentation, the sample custodian immediately notifies the client or the appropriate PACE NE project manager.

The samples are logged into the laboratory system. Each sample group is assigned a unique five digit laboratory number. This number is preprinted on the SRCR sheets. A unique number is assigned to each sample in the group during the receiving process. This Laboratory Number consists of the PACE NE Laboratory Number followed by a numerical suffix serialized to account for the number of samples in a sample group. This laboratory ID is also recorded on the Chain-of-Custody form.

All of this paper work is then used to log the samples into the PACE NE Laboratory Information Management System (LIMS), a computerized management and tracking system. The LIMS generates laboratory worklists of all samples in the system.

6.4 Initiation of Testing Program

Once samples are received and logged into the laboratory system, the Chain-of-Custody, Sample Receipt Condition Report, and any memo or other documentation is placed into a project file created by sample management. If a sample group is a priority or if samples have a short holding time, sample management will immediately provide notification to the appropriate laboratory personnel that samples have arrived and are ready for processing or will deliver the samples directly to the lab. Otherwise, the LIMS automatically produces a daily worklist for each laboratory section listing all samples in the system which need to be processed, the type of quality control samples required, the priority status, holding time and test(s) required.

There is a system for tracking the status and internal chain-of-custody of samples within the laboratory once sample processing begins. The system documents the movement of samples from sample receiving to sample preparation and back and also the movement of processed sample extracts from sample preparation through analysis. The Internal Custody Record is used by sample preparation personnel to locate samples in the sample management storage areas. The transfer from storage to preparation is documented on this sheet. It also serves to document:

- Who removed the sample from storage
- When

- Which samples are associated with a Lab Number
- If the entire sample was entirely consumed
- When and by whom the remainder of sample was returned to storage

The original Internal Custody Record is maintained by Sample Management until the sample disposal date is recorded on it. After sample disposal, the Internal Custody Record is forwarded to the QA department for archiving.

The second system for tracking samples is the LIMS generated worklist. The worklist is generated every night and lists the status for each sample batch/project and for each analytical section. The worklist is distributed to each laboratory section. When the section personnel completes the particular task, they log into the LIMS and complete the information for that batch of samples.

6.5 Sample Disposal

After completion of sample analysis and submission of the analytical report, unused portions of samples are retained by the laboratory for a minimum of 2 weeks. After 2 weeks, samples will be disposed of according to the nature of the samples. The Hazardous Waste Manager receives a copy of the data report and uses that information to select the appropriate waste steam for the samples. The samples are considered hazardous waste and are handled by state and federally licensed hazardous waste disposal firms.

Upon disposal of samples, a computer spreadsheet is maintained by the Hazardous Waste Manager listing the sample number, inherent waste stream and date disposed. This data file is updated on a weekly basis and is kept on file by the Hazardous Waste Manager and Sample Management.

6.6 Subcontracting Analytical Services

Every effort is made to perform chemical analyses for PACE NE clients within the PACE NE laboratory. There are, however, instances where subcontracting of analytical services is necessary. Currently, the following analyses are subcontracted:

- Bacteriological
- Asbestos
- Dioxins
- Radiological
- Total Organic Halides
- NPDES Tin (furnace method 282.1 and 282.2)

When subcontracting becomes necessary, a preliminary verbal communication with an appropriate laboratory is undertaken. The contact and preliminary arrangements and terms of agreement are made between the PACE NE Project Manager and the appropriate subcontract laboratory personnel (i.e., laboratory manager, customer services contact, or the appropriate laboratory section manager). The specific terms of the subcontract laboratory agreement should include (when applicable):

- Method (EPA or otherwise) of analysis
- Number and type of samples expected
- Project specific QA/QC requirements
- Deliverables required
- Applicable laboratory certification status
- Price per analysis
- Turn around time requirements

Chain-of-Custody forms must be generated for samples which require subcontracting to other laboratories. The sample management personnel repackage the samples for shipment, create a transfer chain-of-custody form and record the following information:

- PACE NE Laboratory Number
- Matrix
- Requested analysis
- Special instructions (quick turn around, required detection limits, anything unusual known about the samples or analytical procedure).
- Signature in "Relinquished By"

All subcontracted sample data reports are sent to the PACE NE Project Manager. The Project Manager sends the report to the appropriate PACE NE laboratory manager for review.

Any PACE NE work sent to other labs within the PACE, Inc. network is handled as subcontracted work. All of the conditions and considerations noted in Section 6.6 above apply.

7.0 CALIBRATION PROCEDURES AND FREQUENCY

All instruments and equipment used in the laboratory must follow a well defined calibration routine. Calibration may be accomplished by laboratory personnel using certified reference materials traceable to NIST or EPA certified materials or by external calibration agencies or equipment manufacturers. The discussion presented here is general in nature because the requirements for calibration are instrument (or equipment) and method specific. Details of calibrations can be found in PACE NE Standard Operating Procedures, analytical methods; and operations manuals.

7.1 Standards and Traceability

Analytical standards are prepared from pure compounds or are purchased prepared from reputable vendors. They are used to prepare serial dilutions that are used as calibration and spiking standards. Each laboratory section is responsible for the preparation, storage and disposal of its standards. The preparation information is recorded into section specific Standards Notebooks. The notebooks are where the preparer records all information needed to maintain proper traceability.

Each standard is given an internal identification number. In some instances, particularly with pre-mixed organic standards, the identification number is the solution lot number followed by an PACE NE assigned letter. The preparation of all stock standards shall be documented in a Standards Notebook which is used to record the date of preparation, the analyst, the source of the reference material, amounts used, final volume, etc. and the serial reference number of that stock solution. All standards shall be labelled with the standard serial reference number (small glass ampules), and with the name, concentration, date of preparation and expiration date of the stock standards. All diluted working standards not consumed during an analytical session shall be labelled fully, including the serial reference number of any stock standard used in its preparation.

If no expiration date has been assigned by the manufacturer, then an expiration date of one year from the date of preparation (or the date first opened in the case of sealed ampules) is reported unless degradation prior to this date is observed. To help determine if a standard has degraded, one must note inconsistencies. For instance, very poor recoveries from newly prepared quality control spikes or abnormally low instrument response to a specific standard are indications of possible standard degradation. However, for some standards, degradation is more easily noted. For instance, DDT breaks down to form DDD and DDE. Here one can visually note, on a chromatogram, the degradation of DDT by the increased concentrations of DDD and DDE. If

degradation is observed before the default expiration date, it should be noted in the Standard Notebook for that standard and the standard removed from service. Standards can be held past assigned expiration dates if it can be demonstrated that there has been no degradation.

Before any set of non-CRADA standards can be utilized in a calibration curve they must be verified by a secondary means:

- Analysis of an EPA QC Check Sample, or
- Analysis of an independently prepared check standard.

7.2 General Calibration Procedures

Calibration standards for each parameter are chosen to bracket the expected concentrations of those parameters in the sample, and to operate within the linear response range of the instrument. Samples that fall outside of the calibration range are diluted until bracketed by the calibration standards. Calibration standards are prepared typically at a minimum of three concentration levels, usually chosen at two times, three to five times, and five to ten times the estimated method detection limit plus a calibration blank, with the exception of most organic analyses which do not require a calibration blank. Either an internal standard or external standard quantification technique can be utilized.

Calibration standards are prepared from materials of the highest available purity. To establish instrument calibration, working standards are prepared from more concentrated working stock solutions. All organic standards are refrigerated or frozen. Inorganic standards are refrigerated as necessary. Data regarding their preparation is recorded in the each laboratory sections' Standards Notebook.

Instrumental responses to calibration standards for each parameter are subjected to an appropriate statistical test of fitness (least squares linear regression, quadratic equation, or relative standard deviation of response factors) or as required by the method or QAPP. The calibration must reflect an acceptable correlation of data points or linearity to be acceptable. In cases where the calibration data are outside of these criteria, the analyst must rerun the calibration standards (meeting the same criteria), changing instrumental conditions as necessary.

For analyses which are performed frequently and for which substantial calibration data is available, a complete recalibration is not required each time an analysis is performed providing that the following criterion is met: one calibration standard is analyzed at the

beginning of the analysis which may vary from the expected response (based on the initial, most recent calibration curve) by no more than ±25% or as specified by the method, SOP or QAPP. If this criterion is not met, a complete recalibration is necessary.

During the course of analysis, calibration standards are routinely analyzed to ensure that the instrumental response has not changed. Again the criterion stipulated in each method, or SOP for expected response is used by the analyst to determine whether the instrument must be recalibrated or the instrument conditions further optimized.

The accuracy of prepared standards is periodically checked by comparison with a standard from an independent source.

Certain pieces of equipment such as balances, pH meters, and turbidity meters are normally calibrated with NIST traceable standard reference material.

7.2.1 Analytical Balances

Every six months, calibration of the entire analytical range shall be checked by a qualified service technician. The calibration of each balance is checked each day of use using weights traceable to the National Institute of Standards and Technology (NIST). Calibration weights are Class S or better and are recertified every two years. If balances are calibrated by an external agency, verification of their weights shall be provided. All information pertaining to balance maintenance and calibration is found in the individual balance logbook.

7.2.2 Thermometer

Certified, or reference, thermometers are maintained for checking calibration of working thermometers. Reference thermometers are provided with NIST traceability for initial calibration and are recertified every year with equipment directly traceable to the NIST.

Working thermometers are compared with the reference thermometers every 12 months. Each thermometer is tagged and individually numbered. In addition, working thermometers are visually inspected by laboratory personnel prior to use.

Calibration temperatures and acceptance criteria are based upon the working range of the thermometer and the accuracy required for its use. Laboratory thermometer inventory and calibration data is found in the thermometer logbook.

7.2.3 pH/Electrometer

The meter is calibrated before use each day, and once after each four hours of use using buffer solutions as required by PACE NE SOP QA-800, Use, Calibration, and Maintenance of Equipment - Inorganics Lab.

7.2.4 Spectrophotometer

During use, spectrophotometer performance is checked against CCVs and ICVs. The instrument operating capability is also evaluated every six months by an outside service (QC Services).

7.3 GC/MS Calibration Procedures

The minimum operation necessary to satisfy analytical requirements associated with the determination of organic compounds in water and soil/sediment samples are listed below. The following operations should be performed routinely in the laboratory:

- Documentation of GC/MS mass calibration and abundance pattern
- Documentation of GC/MS response factor stability
- Internal standard response and retention time

Prior to initiating data collection, it is necessary to establish that a given GC/MS meets the standard mass spectral abundance criteria. This is accomplished through the analysis of decafluorotriphenylphosphine (DFTPP) for base/neutral and acid (BNA) compounds or p-bromofluorobenzene (BFB) for volatile compounds. The ion abundance criteria for each calibration compound should be met before any samples, blanks, or standards can be analyzed.

Each GC/MS system used for the analysis of semivolatile organic compounds by EPA methods must be tuned to meet method specific abundance criteria for a 50 nanogram (ng) injection of DFTPP. This criteria must be demonstrated daily. Documentation of the calibration must be provided in the form of a bar graph plot and as a mass listing.

Each GC/MS system used for the analysis of volatile organic compounds by EPA methods must be tuned to meet method specific abundance criteria, typically for a 50 ng injection of BFB. The criteria must be demonstrated daily. Documentation of the calibration should be provided in the form of a bar graph and a mass listing.

Prior to the analysis of samples and after tuning criteria have been met, the GC/MS system must be initially calibrated with a minimum of five concentrations of each compound being analyzed to determine the linearity of response. USEPA criteria specify both the concentration levels for initial calibration and the specific internal standard to be used on a compound-by-compound basis for quantification. The response factor (RF) for each compound at each concentration level is calculated using the following Equation 7.1:

$$RF = \frac{A_x}{A_{is}} * \frac{C_{is}}{C_x} \qquad (7.1)$$

where:

A_x = area of the characteristic ion for the compound to be measured.

A_{is} = area of the characteristic ion for the specific internal standards.

C_{is} = concentration of the internal standard (ng/ul).

C_x = concentration of the compound to be measured (ng/ul)

Using the RF from the initial calibration, the percent relative standard deviation (%RSD) for compounds identified as Calibration Check Compounds is calculated using Equation 7.2:

$$\%RSD = \frac{s}{\bar{x}} \times 100 \qquad (7.2)$$

where:

RSD = relative standard deviation
s = standard deviation of initial five response factors (per compound).
$\bar{x}$ = mean of initial five response factors (per compound).

The %RSD for each individual CCC should be <u>less</u> than 25 % or as specified by the method. This criteria must be met for the initial calibration to be valid.

A calibration standard containing all compounds of interest as well as all required surrogates, is performed each day of analysis. The RF data from the standards is compared each day against the average RF from the initial calibration for a specific instrument. If the response to a calibration check standard differs from the initial calibration by more than ±25% or as specified by the method, then investigation and corrective action must be performed, including a complete recalibration if necessary.

7.4 Gas Chromatography Calibration Procedures

Calibration of a gas chromatograph (GC) for volatiles analyses is similar to GC/MS calibration procedures. Initially, a five point calibration curve, consisting of all compounds of interest at five different concentrations plus a calibration blank, is established to define the linear range of the instrument. The curve is determined to be linear if the correlation coefficient is ≥ 0.99. Linearity may also be determined using response factors. Response factors are calculated for each compound at each concentration level. These RF will be averaged to generate the mean daily RF for each compound over the range of the standard curve. The mean response factor will be used to calculate the sample concentration of the compound of interest. When sample responses exceed the range of the standard curve, the sample will be diluted to fall within the range of the standard curve and be reanalyzed. The results of the daily GC standardization will be tabulated and filed with the corresponding sample analyses. Daily full calibration is not necessary if a calibration check standard validates the initial calibration curve. If the response to a calibration check standard differs from the initial calibration by more than ±15% for any analyte being quantitated or as specified by the method, then investigation and corrective action will be performed, including complete recalibration, if necessary.

Initial Calibration of a GC for semivolatile compound analyses (pesticides/herbicides) typically involves a five point calibration curve, consisting of all compounds of interest at five different concentrations to define the working range. Using analyte peak height or peak area, a quadratic curve is applied and extrapolated through the origin. A correlation coefficient of the calibration data should be 0.99 or better for the calibration to be used for quantitation. Continuing Calibration is checked every ten samples or 24 hours, whichever comes first after the initial calibration by analyzing mid-point calibration standards. Each analyte of interest in the continuing calibration must be within 15% of the peak height observed in the initial calibration. For certain multi-peak compounds, the average result for 5 peak heights must be within 15% of the average of the same 5 peak heights observed in the initial calibration.

7.5 Calibration of Inductively Coupled Argon Plasma Spectrophotometer (ICP) and Atomic Absorption Spectrophotometer (AAS)

The ICP and AAS are standardized for the metal of interest by the analysis of a set of calibration standards prepared by diluting a stock solution of known concentration. Working standards are prepared by dilution of the stock standard. For the AAS, the concentration of the calibration standards is chosen so as to cover the working range of the instrument. For ICP, a standard is analyzed as a sample to determine the upper limit of the calibration. Subsequently, all sample measurements are made within this working range. Once the working standards are prepared, they are analyzed on the ICP or AAS and the instrument response is calibrated to provide a direct readout in concentration.

The calibration is accomplished by entering the metal concentration equivalent to the readout in absorbance units (or emission intensity) during analysis of the working standards.

Once the instrument has been initially calibrated, the analysis of the working standards is repeated during sample analysis to standardize instrument response during analysis and to confirm the calibration settings. A typical analysis sequence is presented below.

- Working standards are prepared by dilution of a stock standard solution of the metal of interest.
- A calibration curve within the working range of the instrument is established by analysis of three to five working standards.
- An independent standard is analyzed to confirm the calibration settings. If the calibration settings are not confirmed, the instrument is recalibrated.
- The samples are analyzed for the metal of interest.
- During sample analysis, a check standard is analyzed to monitor instrument stability. If the analysis indicates that instrument calibration has changed by more than $\pm 10\%$ for ICP or more than $\pm 20\%$ for AAS, the instrument is recalibrated and the analysis is repeated.
- Following completion of the sample analyses, the check standard is reanalyzed to confirm calibration settings. If calibration settings are confirmed, the analysis is completed. However, if the calibration settings are not confirmed, the problem is corrected, and the analyses are repeated.

Written records of all calibrations shall be kept in the appropriate instrument logbook.

8.0 ANALYTICAL PROCEDURES

PACE NE Laboratories are capable of analyzing the full range of environmental samples from all media, including surface and groundwater, soil, sediment, tissue, and waste. Refer to Table 8-2 for a listing of specific PACE NE analytical capabilities. The methodologies generally employed constitute the most recent guidance from agencies such as EPA, ASTM, USGS, NIOSH and in certain instances, state regulatory agencies. In some situations, PACE NE develops and validates methodologies which are more applicable to a specific problem or objective.

Analytical procedures are detailed descriptions of any and all processing, preparation and analysis of samples in the laboratory. In some instances, data format, presentation and delivery are also described. All analytical procedures shall be conducted in strict adherence with written Standard Operating Procedures manuals which have been reviewed and approved by the Laboratory Manager(s)/Technical Director(s), the PACE NE QA Officer and the PACE NE Regional Director. Documents from which SOPs are developed include the references listed in Table 8-1. Additional SOPs may be adapted from other sources or generated in-house as project needs require.

8.1 Analytical Methods

Numerous sources of information are available to offer guidance in analytical methods. Selection of the appropriate method is dependent upon data usage and the regulatory requirements during the analysis. Table 8-1 describes the analytical references routinely used by PACE NE Laboratories. PACE NE may modify existing methods based on the following considerations: 1) in order to meet project specific objectives; 2) in order to incorporate modifications or improvements in analytical technology; 3) in order to comply with changing regulations and requirements; 4) in order to address unusual matrices not covered in available methods.

PACE NE will make every effort to disclose to its clients any instances in which modified methods are being used in the analysis of samples.

8.2 Method Validation

When an established method is first used by the laboratory or when the laboratory develops a method, the laboratory establishes the validity of the method prior to applying it to client samples. Method validity is established by meeting certain criteria for precision and accuracy.

The design of the validation study will vary with the nature of the matrix, parameters and requirements of the client. However, in all cases, the laboratory demonstrates that adequate and reproducible recoveries are attainable. The precision of the test method is measured as the relative standard deviation for a minimum of four (4) replicate analyses of the lowest concentration recovery sample.

Accuracy of the test method is established as the relative standard deviation of the average measurement of each recovery sample (at a specific concentration). Design of validation studies incorporates several recovery samples at each concentration level. Thus, it is feasible to determine the test method accuracy at each concentration level, if significant variances occur with concentration.

8.3 Method Detection Limits

When an analytical procedure does not detect a parameter of interest, it is important to know what the lower limit of detection is for that particular method and sample matrix. The laboratory endeavors to prevent matrix interferences from substantially reducing analytical sensitivity, thereby raising the analytical detection limit. When matrix interferences are present, various clean-up techniques may be employed to reduce or eliminate them.

Method detection limit studies are performed for each method in use at least annually and after any procedural or configurational change.

Method detection limits may be determined using replicate spiked laboratory water samples. A minimum of seven aliquots of a sample spiked for the purpose are processed through the entire analytical method. The concentration of the detection limit sample should be between 2 and 5 times the anticipated detection limit.

The laboratory calculates the detection limit as 3.143 times the standard deviation of replicate measurements of the spiked samples. The reader is referred to 40 CFR Part 136, Appendix B for further discussion.

IT IS IMPERATIVE TO NOTE THAT METHOD DETECTION LIMITS ARE HIGHLY MATRIX DEPENDENT. THE LIMITS LISTED IN THE CITED METHODS ARE FOR GUIDANCE AND MAY NOT ALWAYS BE ACHIEVABLE.

8.4 Compliance

8.4.1 Definition - Compliance is the proper execution of recognized, documented procedures which are either approved or required. Adherence to these procedures is required in order to provide data products acceptable to a regulatory body of competent jurisdiction in a specific regulatory context. Compliance is separate from, but not inconsistent with, technical scientific quality. PACE NE accepts compliance as part of the PACE corporate definition of quality: "Quality is the *fulfillment of expectations and needs* in all activities, demonstrated by the satisfaction of those we serve." PACE NE understands that the expectations of our clients commonly include the assumption that data and reports will satisfy a regulatory purpose and will be found acceptable *and compliant* with regulatory requirements for the performance of tests and generation of data.

8.4.2 Understanding the Regulatory Framework - Compliance is not likely to be achieved in the absence of an understanding of the regulatory framework. PACE NE will attempt to ascertain, prior to beginning a project, what regulatory jurisdiction (USEPA, NJDEPE, etc.) pertains to a project; within the regulatory jurisdiction, what body of regulation is meant to be satisfied (RCRA, SDWA, ECRA, 21E, etc.); and finally, within this context, what protocols are required/expected (CLP, AFCEE, NEESA, ASP, etc.). PACE NE will work with its clients to come to a mutual understanding of all requirements.

8.4.3 Commitment - Clients may, but often do not, fully understand their compliance needs. Clients may sometimes fail to communicate their compliance requirements to PACE NE. Nevertheless, PACE NE, Inc., in defining quality as in 8.4.1 above, has accepted much responsibility for compliance.

PACE NE makes the following commitments to its clients:

1) PACE NE will proactively attempt to identify and understand the regulatory context of clients' needs.

2) PACE NE will strive to be expert in understanding and executing the regulatory requirements for compliance.

3) PACE NE will identify and disclose to clients instances of non-compliance in a forthright fashion.

8.4.4 Resolving Compliance Cantradictions and Hierarchies - It is a common occurrence that multiple regulatory juristictions overlap in a specific case. This causes uncertainty or even contradictions to arise in a work plan. PACE NE will make every effort to detect such inconsistencies, and will communicate them to clients so that an informed decision can be made by the client regarding execution

of the project. Similarly, methods and protocols will often be prescribed in a scope of work or QAPP which either will not achieve stated or implied DQOs or which are in conflict with the regulatory requirements. PACE NE will attempt to detect these inconsistencies, and upon detection, disclose same to our client. PACE NE voluntarily accepts a responsibility to provide advice to clients, however, **the primary responsibility for this issue remains with the client.**

8.4.5 Disclosure of Noncompliance - As stated previously, it is PACE NE policy to disclose in a forthright manner any detected noncompliance that may effect the usability of data produced by PACE NE. It is not within our expertise to predict the manner in which a specific regulator or regulatory body will interpret the rules governing analysis; PACE NE is unable to guarantee compliance. It is PACE NE policy that our responsibility begins with a bona fide and competent attempt to evaluate potential compliance issues and ends with disclosure of any findings that may be useful to our client in their making the final judgement.

TABLE 8-1

ANALYTICAL PROTOCOLS

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act." Federal Register, 40 CFR Part 136, October 26, 1984.
- "Test Methods for Evaluating Solid Wastes: Physical/Chemical Methods." SW-846. 2nd edition, 1982 (revised 1984), 3rd edition, 1986, Office of Solid Waste and Emergency Response, U.S. EPA.
- "Methods for Chemical Analysis of Water and Wastes", EPA 600/4-79-020, 1979 Revised 1983, U.S. EPA.
- U.S. EPA Contract Laboratory Program Statement of Work for Organic Analysis, SOW 2/88, and OLM01.8, 8/91.
- U.S. EPA Contract Laboratory Program Statement of Work for Inorganic Analysis, SOW No. 788, ILM01.0, 3/90 and ILM02.0.
- "Standard Methods for the Examination of Water and Wastewater", 15th, 16th and 17th editions, 1980, 1985, 1989. APHA-AWWA-WPCF.
- "Annual Book of ASTM Standards", Section 4: Construction, Volume 04.04: Soil and Rock; Building Stones, American Society for Testing and Materials, 1987.
- "Annual Book of ASTM Standards", Section 11: Water and Environmental Technology, American Society for Testing and Materials, 1987.
- "NIOSH Manual of Analytical Methods", Third Edition, 1984, U.S. Department of Health and Human Services, National Institute for Occupational Safety and Health.
- "Methods for the Determination of Organic Compounds in Finished Drinking Water and Raw Source Water", U.S. EPA, Environmental Monitoring and Support Laboratory - Cincinnati (September 1986).
- New York State Department of Environmental Conservation. Analytical Services Protocol, September, 1989.

TABLE 8-2

PACE NE ANALYTICAL CAPABILITIES
ORGANIC ANALYSES

Analyte	EPA Methods	
Volatile Organic Compounds		
GC/MS	624	8240
GC/MS + 15 Peaks*	624	8240
CLP Volatiles Analysis and Deliverables	3/91 SOW	3/91 SOW
GC/MS Low Detection Limits	624	8240
GC/MS Drinking Water Volatiles	524.1	NA
GC/MS Drinking Water VOA (Extended List)	524.2	NA
GC Drinking Water Halogenated Compounds	502.1	NA
GC Drinking Water Aromatic Compounds	503.1	NA
GC Drinking Water VOA(Combined 502.1 & 503.1)	502.2	NA
Purgeable Halocarbons	601	8010
Purgeable Aromatics	602	8020
Combined Purgeables	601 & 602	8010 & 8020
Acrolein, Acrylonitrile	603	8030
Non-halogenated Volatiles (Partial List)	8015	8015
Base Neutral/Acid Extractables (HSL)	625	8270
Base Neutral/Acid Extractables + 25 Peaks*	625	8270
Acid Extractables Only (HSL)	625	8270
Acid Extractables Only + 10 Peaks*	625	8270
Base Neutral Extractables Only (HSL)	625	8270
Base Neutral Extractables Only + 15 Peaks*	625	8270
CLP Semivolatiles Analysis and Deliverables	3/91 SOW	3/91 SOW
Polynuclear Aromatics (PNA) GC/MS	625	8270
Polynuclear Aromatics (PNA) GC	610	8100

* Tentative identification via computer search of specified number of non-target peaks.

TABLE 8-2, continued

ORGANIC ANALYSES (continued)

Analyte	EPA Methods	
Organochlorine Pesticides & PCBs	608	8080
CLP Organochlorine Pesticides & PCBs	3/91 SOW	3/91 SOW
Organochlorine Pesticides & PCBs (Tissue)	608	8080
Organophosphorus Pesticides	614	8140
Chlorinated Phenoxy Herbicides	8150	
PCBs in oils	ASTM	
PCBs wipes/filters	ASTM	
Petroleum Hydrocarbons, Extractables (water/soil, tissues)	Capillary GC/FID	
Petroleum Hydrocarbons, Purgeables	Modified 8015	
Gasoline Hydrocarbons BTEX(+MTBE)	602	8020
Compositing		

TABLE 8-2, continued

WASTE CHARACTERIZATION

Analyte		EPA Methods
EP Toxicity	Extraction	1310/1330
	Metals in Extract	
	Arsenic	6010
	Barium	6010
	Cadmium	6010
	Chromium	6010
	Lead	6010
	Mercury	7470
	Selenium	6010
	Silver	6010
	Pesticides in Extract	8080
	Endrin	
	Lindane	
	Methoxychlor	
	Toxaphene	
	Herbicides in Extract	8150
	2,4-D	
	2,4,5-TP	
Corrosivity	pH	9040/9045
Reactivity	Releasable Cyanide	SW 846 7.3.3.2
	Releasable Sulfide	SW 846 7.3.4.1
	Releasable Cyanide & Sulfide	
	Water Reactivity	N/A
Ignitability		1010

TABLE 8-2, continued

TCLP ANALYSES

Quality Assurance section of the method requires that a matrix spike be performed for each sample type. A determination as to whether a spike is needed must be made prior to submitting the sample. Matrix spikes are made at five times the method detection limit or at the regulatory limit unless other levels are specified in advance.

Matrix spike for pesticides requires that single component pesticides be analyzed separately from multicomponent pesticides (toxaphene). Therefore, two matrix spikes are required and are reflected in the matrix spike analysis.

Samples containing free liquid that is not miscible with the TCLP extract require that both the free liquid and the extract be analyzed separately and that the results be combined mathematically. An additional charge is required for the analysis of the free liquid or of multiple phases. If a matrix spike is required, only the TCLP extract is spiked.

Analyte		EPA Methods
Extraction for Volatiles (ZHE)		1311
Extraction for Metals, Semivolatiles, Pesticides & Herbicides		1311
Volatiles	benzene	8240
	carbon tetrachloride	
	chlorobenzene	
	chloroform	
	1,2-dichloroethane	
	1,1-dichloroethylene	
	methyl ethyl ketone	
	tetrachloroethylene	
	trichloroethylene	
	vinyl chloride	

TABLE 8-2, continued

TCLP ANALYSES (continued)

Analyte		EPA Methods
Semi-Volatiles	*pyridine*	8270
	m-cresol (3-methyl phenol)	
	o-cresol (2-methyl phenol)	
	p-cresol (4-methyl phenol)	
	1,4-dichlorobenzene	
	2,4-dinitrotoluene	
	hexachloro-1,3-butadiene	
	hexachlorobenzene	
	hexachloroethane	
	nitrobenzene	
	pentachlorophenol	
	2,4,5-trichlorophenol	
	2,4,6-trichlorophenol	
Pesticides	chlordane	8080
	endrin	
	heptachlor	
	heptachlor epoxide	
	lindane	
	methoxychlor	
	toxaphene	
Herbicides	2,4-dichlorophenoxyacetic acid	8150
	2,4-5-trichlorophenoxypropionic acid	
Metals	As, Ba, Cd, Cr, Pb, Se, Ag	6010
	Hg	7470

TABLE 8-2, continued
METALS ANALYSES

Analyte	Required Digest	EPA Methods
Aluminum	3010/3050	200.7 or 6010
Antimony	3010/3050	200.7 or 6010
Antimony (furnace)	3010/3050	204.2 or 7041
Arsenic (furnace)	7060/3050	206.2 or 7060
Barium	3010/3050	200.7 or 6010
Beryllium	3010/3050	200.7 or 6010
Boron	3010/3050	200.7 or 6010
Cadmium	3010/3050	200.7 or 6010
Cadmium (furnace)	3020/3050	213.2 or 7131
Calcium	3010/3050	200.7 or 6010
Chromium	3010/3050	200.7 or 6010
Chromium (furnace)	3020/3050	218.2 or 7191
Cobalt	3010/3050	200.7 or 6010
Copper	3010/3050	200.7 or 6010
Iron	3010/3050	200.7 or 6010
Lead	3010/3050	200.7 or 6010
Lead (furnace)	3020/3050	239.2 or 7421
Magnesium	3010/3050	200.7 or 6010
Manganese	3010/3050	200.7 or 6010
Mercury (cold vapor)	7470/7471	245.1 or 7470/7471
Molybdenum	3010/3050	200.7 or 6010
Nickel	3010/3050	200.7 or 6010
Potassium	3010/3050	200.7 or 6010
Selenium (furnace)	7740/3050	270.2 or 7740
Silver	3010/3050	200.7 or 6010
Sodium	3010/3050	200.7 or 6010
Strontium	3010/3050	200.7 or 6010
Thallium (furnace)	3020/3050	279.1 or 7841
Tin	3010/3050	200.7 or 6010
Titanium	3010/3050	200.7 or 6010
Vanadium	3010/3050	200.7 or 6010
Zinc	3010/3050	200.7 or 6010
Packages Available	CLP Deliverables-23 Metals 13 Priority Pollutant Metals 8 Safe Drinking Water Act Metals 23 HSL Metals-Commercial Deliverables	
Digestions		
Water	3010,3020,7060,7740	
Soil/ICP	3050	
Soil/GFAA	3050	
Tissues/ICP	3050 Modified	
Tissues/GFAA	3050 Modified	
Mercury-water/soil	7470/7471	
Dissolution (Oils)	3040	

TABLE 8-2, continued

INORGANIC ANALYSES

Analyte	Methods
Acidity	EPA 305.1
Alkalinity	EPA 310.1
Bromide	EPA 300.0
Biochemical Oxygen Demand	EPA 405.1
Bicarbonate	APHA 2320
Carbonate	APHA 2320
Bicarbonate & Carbonate	APHA 2320
Carbon - Total Inorganic (water)	EPA 415.1
- Total Organic (water)	EPA 415.1
- Total (soil)	
- Total Organic (soil)	
Cation Exchange Capacity (soil)	EPA 9080
Chemical Oxygen Demand	
Detection limit of 20 mg/L	EPA 410.4
Detection limit of 5 mg/L	EPA 410.4/410.2
Chloride	EPA 300.0 or 325.1
Chlorine, Total Residual	APHA 4500-CLG.
Coliform Bacteria - Total	APHA 9222B
- Fecal	APHA 9222D
Color	
Chromium - Hexavalent	SW 7196
Cyanide - Total	EPA 335.2
Cyanide - Total Ammenable	EPA 335.1
Cyanide, WAD	APHA 4500-CNI.
Density	
Fluoride EPA 340.2	
Formaldehyde	NIOSH 3500
Hardness (Ca, Mg by calculation)	EPA 200.7 and SM 314 B
Calculation only	
Halides, Total Organic (TOX) - Water	9020
- Solids/Oils	
Lime Equivalence	AOAC 1.004-6
Nitrogen - Ammonia	EPA 350.1 or 350.3
- Nitrate plus Nitrite Nitrogen (combined)	353.2
- Nitrate	EPA 300.0 or 353.2
- Nitrite	EPA 300.0 or 353.2
- Total Kjeldahl	EPA 351.2 or 353.3
- Total Organic-Calculation Fee (requires analysis of TKN & NH3)	

TABLE 8-2, continued

INORGANIC ANALYSES (continued)

Analyte	Methods
Oil & Grease (Gravimetric or IR)	EPA 413.1/413.2
Oxidation Reduction Potential	
Free Liquid Content (Paint Filter Test)	EPA 9095
Total Petroleum Hydrocarbon IR	EPA 418.1
pH	EPA 150.1
Phenols - Total	EPA 420.3
Phosphorus - Total	EPA 365.3 or 365.4
- Ortho	EPA 365.1 or 365.3
Silicate - Reactive	EPA 370.1
Solids - Total	EPA 160.3
- Suspended	EPA 160.2
- Dissolved	EPA 160.1
- Volatile	EPA 160.4
- Suspended Volatile	EPA 160.2 or 160.4
- Settleable	EPA 160.5
Specific Conductance	EPA 120.1
Specific Gravity	EPA/COE 1981
Sulfate	EPA 300.0 or 375.2
Sulfide	EPA 376.2
Sulfite	EPA 377.1
Surfactants - MBAS	EPA 425.1
Tannins and Lignins (as tannic acid)	APHA 5550B
Turbidity	EPA 180.1
Sample Prep:	
Compositing	
Filtration	APHA 3030B

TABLE 8-2, continued

APPENDIX NINE ANALYSES

Appendix IX is derived from Appendix VIII Hazardous Constituents. These parameter lists are intended to apply to groundwater monitoring at hazardous waste storage and disposal sites and are applied to uncontrolled site investigations and remediations.

Analyte	EPA Methods
Volatile Organics (extended list 19 Compounds)	8240
Acrolein and Acrylonitrile	8030
Acid and Base Neutrals (extended list 48 Compounds)	3550/8270
Dioxins/2378 TCDDD	8280
Pesticides and PCB's	3540/8080
Herbicides	8150
Total Cyanide	9010
Hydrogen Sulfide	376.1
pH	150.1
17 Metals	
Arsenic	7060
Antimony	6010
Barium	6010
Beryllium	6010
Cadmium	6010
Cobalt	6010
Chromium	6010
Copper	6010
Mercury	7470
Nickel	6010
Lead	7421
Selenium	7740
Silver	6010
Tin	6010
Thallium	7841
Vanadium	6010
Zinc	6010
Metals Digest	3010,3020,7060,7470

TABLE 8-2, continued

FORM 2C ANALYSES FOR NPDES PERMITS

Analyte	Methods
Part A	
Biochemical Oxygen Demand	EPA 405.1
Chemical Oxygen Demand	EPA 410.4
Total Organic Carbon	EPA 415.1
Total Suspended Solids	EPA 160.1
Ammonia as Nitrogen	EPA 350.2/350.3 or 350.2/350.1
pH	EPA 150.1
Part B	
Total Residual Chlorine	EPA 330.5
Color	EAP 110.2
Fecal Coliform	APHA 9222D
Fluoride	EAP 340.1/340.2
Nitrate	EPA 353.2 or 300.0
Nitrite	EPA 353.2 or 300.0
Total Organic Nitrogen	350.2/350.3/351.3 or 350.2/350.1/351.2
Oil and Grease	EPA 413.1 or 413.2
Total Phosphorus	EPA 365.1 or 365.4
Alpha	
Beta	
Radium	
Sulfate	EPA 300.0
Sulfide	EPA 376.1
Sulfite	EPA 377.1
Surfactants	EPA 425.1
Aluminum	EPA 200.7
Barium	EPA 200.7
Boron	EPA 200.7
Cobalt	EPA 200.7
Iron	EPA 200.7
Magnesium	EPA 200.7
Molybdenum	EPA 200.7
Manganese	EPA 200.7
Tin	EPA 200.7
Titanium	EPA 200.7
Metals Digestion	(3010)

TABLE 8-2, continued

FORM 2C ANALYSES FOR NPDES PERMITS (continued)

Analyte	Methods
Part C	
Antimony	EPA 200.7
Arsenic	EPA 206.2
Beryllium	EPA 200.7
Cadmium	EPA 200.7
Chromium	EPA 200.7
Copper	EPA 200.7
Lead	EPA 239.2
Mercury	EPA 245.1
Nickel	EPA 200.7
Selenium	EPA 270.2
Silver	EPA 200.7
Thallium	EPA 279.1
Zinc	EPA 200.7
Total Cyanide	EPA 335.2 or 335.3
Total Phenols	EPA 420.3
Dioxin	EPA 8280
Volatile Organic Compounds	EPA 624
Acid and Base Neutral Compounds	EPA 625
Pesticides and PCB's	EPA 608
Metals Digestions	(3010,3020,7060)

TABLE 8-2, continued

NON-ROUTINE ANALYSES

PACE New England has the capability to perform the following analyses upon request. Turnaround time is subject to instrument availability and prices are quoted based on analyst and instrument time required and quantity of samples submitted.

Analysis	Methods
BTU	ASTM D 240
Chlorine (%)	ASTM D 129-64
Sulfur (%)	ASTM D 129-64
HalogenS	ASTM D 808
Viscosity	ASTM D 445
Flash point	ASTM D 93-77
API Gravity	ASTM D 287
Ash Content	ASTM D 482-87
pH	SW846 9045
Free Liquids	(Paint Filter)
% Moisture	APHA 2540G
% Sand, Silt, Clay	EPA/CE-81-1
Sieve Analysis (6 fractions >8mm to <2mm)	EPA/CE-81-1
Particle Size (19 fractions >8mm to .02mm)	EPA/CE-81-1
Diquat, Paraquat by HPLC	EPA 549
Carbamate Pesticides by HPLC	EPA 531
PNA by HPLC	EPA 8310
Ethylene Glycol by HPLC	
Nitrogen-Phosphorus Pesticides by GC	EPA 507
EDB & DBCP by GC	EPA 504
GC Direct Inject for Alcohols	
Volatile Air Analysis in Tedlar Bags by GC or GC/MS	
Calibrated Volatile Non-target Compounds by GC/MS	
Method Development for any Organic Compounds by HPLC, GC/MS, or GC	
Non-routine Furnace or ICP Metals	
Bromide by Ion Chromatagraph	EPA 300.0
Formaldehyde by HPLC	EPA 8315
Nitroaromatic Explosives by HPLC	EPA 8330

9.0 DATA REDUCTION, VALIDATION AND REPORTING

All analytical data generated within the PACE NE Laboratories undergo a well-defined, well-documented multi-tier review process before being reported to the client.

9.1 Data Reduction

When primary analytical data, otherwise known as "raw data", are manually generated, the data are recorded either in bound Logbooks with prenumbered pages or on preprinted forms. Entries are made in black ink and are initialled and dated by the individual who makes the entry. It is acceptable to initial and date once for an entire page. Errors are corrected by drawing a single line through the entry; this change is initialled and dated by the individual who makes the change. The reason for the change is indicated either by description or by error code (see Figure 9-1). Raw data may not be obscured in any way. The use of white-out is prohibited on all raw data, including instrumental hardcopy.

The analyst who completes the analysis assembles all relevant raw data and results together with chromatograms, strip chart recordings, instrument settings and other information essential to data interpretation. For data which are reduced by manual calculations, the calculations are documented in a laboratory notebook or on an analyst's worksheet. The results are transferred to a standard laboratory reporting form which has been approved by the Group Supervisor and Lab Manager. Reporting forms include at a minimum the sample identification number, the date analyzed, the result expressed per unit volume, the method reference and the analyst's initials.

9.2 Data Validation

The analyst initiates the data validation process by reviewing and accepting the data. The completed data package is then sent to the Group Supervisor. The Group Supervisor provides a technical assessment of the data package and technical review for accuracy according to methods employed and laboratory protocols. Group Supervisors also review analyst generated calculations.

For data which are reduced via computer, calculations are checked by the analyst (or designee) assigned to this task at a frequency designed to assure that the data manipulations are valid. This data validation step is documented by the analysts' initials on the hardcopy of the raw data. The results are either manually transferred to a standard reporting form or reported via computer generation of forms.

The Group Supervisor submits the validated data package, incl[illegible]ing the re[illegible]ng forms and the appropriate laboratory control sheets, to the [illegible]orator[illegible]nager wno reviews the package for QC requirements and completeness. [illegible]any erro[illegible]re d[illegible]vered, the entire package shall be returned to the Group Supervisor for full reworki[illegible]

When accepted, the Laboratory Manager assembles the data wi[illegible]ther data from the sample set, drafts any narrative comments if required by the Qua[illegible] Assur[illegible]e Project Plan, and forwards the report and the data package to the Reporting Dep[illegible]ent.

The Reporting Department assembles and types the [illegible]nal r[illegible] for transcription [illegible]rors, and provides the final report to the [illegible]ratory Ma[illegible] Director for [illegible]al signature.

The Laboratory Manger/Technical Director examines the repor[illegible] method appropriateness, detection limits and whether or not QC criteria were sati[illegible]d. Any deviations from the referenced methods are checked for documentation and validity, and QC corrective actions are reviewed for successful resolution. T[illegible] Laboratory Manager/Technical Director signs the completed reports prior [illegible]eir r[illegible]ase.

The Laboratory Manager/Technical Director may delegate the final review and signing of reports to the Regional Director, as necessary.

9.3 Data Report

All data segments pertaining to a particular PACE NE Laboratory Number are channeled to the Reporting Department for assembly into the final repor[illegible] format and generation of the analytical narrative. All points mentioned during technical and QC [illegible]view are included in the narrative if it is deemed to impact the quality of the d[illegible]

The final report is given to the Laboratory Manager/Tech[illegible] [illegible]or final review and release. After verifying the report's complet[illegible]y, the Technical Director signs the cover letter indicating acceptance o[illegible]

The standard commercial report to the client consists of the following sections:

1) the narrative
2) sample results
3) Chain-of-Custody forms

The narrative briefly describes the condition of the samples upon receipt, sample holding time performance, instrument calibration information, and the quality control results. Any discrepancies discovered and matrix problems are also addressed in this section.

The sample results are tabulated. PACE NE number, client identification, and data analyzed are presented along with the observed concentrations for each parameter analyzed and detection limits.

9.4 Data Archive

Each data report which supports the analytical process for all samples received by the laboratory is thoroughly reviewed for completeness and accuracy. After the technical review it is routed to the Reporting Department for assembling the final report for submission to the client. The report is approved, signed, and submitted.

One copy of the report remains with all the raw data which is stored in the data archives under the control of the QA Department.

The Data Archivist has oversight responsibility for the data archive ensuring the continued integrity of all documentation generated in support of laboratory analyses.

The archive room is a secure storage area with limited access to non-authorized personnel. Sign-out procedures are in place where every document removed from the archive room must be electronically signed out by a member of the QA Department.

A copy of the report is forwarded to the Hazardous Waste Manager for use in characterizing the samples for ultimate disposal.

TABLE 9-1.

Error Codes

1 (or E1)	Misspelled
2 (or E2)	Mathematical Error
3 (or E3)	Wrong Entry
4 (or E4)	Transposition or Sequencing Erro
5 (or E5)	Transcription Change (copy error)
6 (or E6)	Procedural Change
7 (or E7)	Wrong Conclusion
8 (or E8)	Illegible Entry
9 (or E9)	Unnecessary Entry
10 (or E10)	Footnoted Explanation
11 (or E11)	Additional Comment
12 (or E12)	Instrumentation Error/Failure

10.0 RECORDS MANAGEMENT

Records are the means by which an organization documents its operations and activities. They are an integral part of the Quality Assurance program since they provide documented evidence for program functionality and necessary information for performance evaluation and quality assurance audits.

10.1 Standard Operating Procedures

Standard Operating Procedures (SOPs) are written for specific procedures or operations. Complex tasks of inspection, testing, calibration, monitoring, maintenance, data handling, and quality control as well as methods utilized in the laboratory are specified and documented by SOPs.

As a minimum requirement, each SOP must include a title, the purpose or applicability, list of materials or references, and detailed procedures. SOPs are updated when necessary. Each has an PACE NE reference number and revision data at the upper right corner of each page. More detailed information regarding SOPs can be found in Section 12.

All personnel are required to follow these documents when a specific operation or method is being utilized. It is the responsibility of the supervisor to make sure that employees are aware of and follow the SOPs. Any suggestions for additional SOPs or changes to existing SOPs can be directed to the QA Department.

10.2 Sample Tracking

Samples are tracked from the time they are received, through storage, preparation, analysis, and final disposition.

Proper sample identification must be established during sample collection. This information must be clearly and permanently written on a label and attached to the sample. In addition, a Chain-of-Custody must be initiated with the appropriate information recorded. Samples should also be properly preserved and stored.

Sample Management Personnel verifies the samples' integrity as they are unpacked. The PACE NE Project Manager and/or the Client are notified of samples that are broken or have not been properly stored or preserved. The sample identification label must also be checked against Chain-of-Custody identification. Any discrepancies must be verified

by the Client or sampler. All these checks and any discrepancies or changes must be documented on the Sample Receiving documentation.

The Sample Management Personnel assign each sample a sequential laboratory identification number, which is written on the sample container and recorded onto the Chain-of-Custody. Samples are properly stored in their respective storage refrigerators.

Upon receipt the Sample Management Personnel also initiates an Internal Custody Record for the sample set. This sheet is used to document sample removal from and return to sample storage. The final disposition of a sample is documented on the hazardous waste disposal spreadsheet and on the internal sample custody record.

10.2.1 Chain-of-Custody

The Chain-of-Custody Form traces the possession of a sample from the time the sample is obtained in the field through analysis in the laboratory. To initiate a Chain-of-Custody, the field sampler must fill in the appropriate information: Client or Project Name, Signature of Sampler, Sample Identification, Date and Time Sampled, Type of Sample, and Analysis Requested. After the sample is brought into the laboratory, sample breakage, preservation, and identification are checked. Any inconsistencies are noted in the sample management documentation. For samples accepted into the laboratory, the Sample Management Personnel record the assigned lab sample number for each sample onto the Chain-of-Custody form, signs in the space marked "Received for Laboratory" and records the date and time received.

10.2.2 Internal Sample Custody Record

The Internal Custody Record is created at the time of sample receipt and is a mechanism for tracking samples from sample management to sample preparation and back. When laboratory personnel remove samples from sample storage, it is recorded on the Internal Custody Record. Upon returning any remaining sample, the individual records the date in the "Returned By" box for the appropriate sample. The original Internal Custody Record is archived by the QA department.

10.3 Standards

Standards preparation is documented at PACE NE in a Standards Logbook maintained by each laboratory. These logbooks are where the preparer records all information

needed to maintain proper traceability. More complete information regarding standards is provided in Section 7.1.

10.4 Maintenance Logbooks

Maintenance Logbooks are kept for each instrument. Each logbook is unique to its instrument. In the logbook, an analyst records initial instrument setup, routine preventive maintenance, and instrumental malfunctions, dates taken in and out of service, and resolutions. Instrument logs not only describe the instrument's history, but can be helpful when troubleshooting.

10.5 Data Report/Raw Data Package

A Data Report contains the results of analyses as presented to the Client. There is a narrative describing the general condition of the samples and an overview of the analyses. For more information see Section 9.3.

The Raw Data Package contains information needed to reconstruct how the analysis of a batch of samples including QA/QC results data were derived. Information such as inorganic or organic preparation, chromatograms or strip chart recording, and regression analysis data are usually included.

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11.0 QUALITY CONTROL

A quality control program is a systematic process that controls the validity of analytical results by measuring the accuracy and precision of each method and matrix, developing expected control limits, using these limits to detect errors or out-of-control events, and requiring corrective action techniques to prevent or minimize the recurrence of these events.

11.1 Accuracy and Precision Measurements

The results of quality control samples created in the laboratory represent estimates of accuracy and precision for the preparation and analysis steps of sample handling. This section describes the quality control information provided by each of these analytical measurements. Information on the procedures to follow in preparation of the samples or spiking solutions is described for each method and matrix in the respective method Standard Operating Procedure.

Method Blank

A method blank is a volume of deionized, distilled laboratory water for water samples, or a purified solid matrix for soil/sediment samples, carried through the entire analytical procedure. The volume or weight of the blank must be approximately equal to the sample volume or weight processed. A method blank is performed with each batch of samples or one with every 20 field samples whichever is more frequent. Analysis of the blank verifies that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware are known and minimized. Optimally, a method blank should contain no greater than five times (5X) the method detection limit for common laboratory solvents and phthalate esters; less than the detection limit for all other parameters unless otherwise specified in the method or project QA plan. Results of method blank analyses are maintained with other QC data in the respective laboratories. If requested by the client, this data will be included in the report.

Accuracy Measurements

Laboratory Control Samples (LCS), consist of aliquots of ideal matrices (water, sand, etc.) spiked with analytes of interest. LCS's for methods with extensive lists of analytes that may interfere with one another may include a limited number of analytes, but the analytes included must be representative of as many analytes as is practical. In the case of metals analysis, all analytes of interest must be included.

Laboratory pure water is used to prepare most LCS's for methods for analysis of water. Highly characterized solids, where available, are used for LCS's for methods for analysis of solids. Where no such solid LCS is available, spiked laboratory pure water or spiked reagent blanks may be substituted. LCS's provide an estimate of bias based on recovery of the compounds from a clean, control matrix. They provide evidence that the laboratory is performing the method within accepted guidelines without potential non-matrix interferences. They are prepared in duplicate at a rate of one set per twenty samples or one set per 14 days whichever is more frequent.

For tests that are performed infrequently, an LCS and its duplicate shall be analyzed at least monthly if the number of samples is less than 20. This monthly requirement shall NOT apply to low-volume tests for which state certification is not sought, or for tests expected to be performed solely as part of a special project, or for tests involving study specific matrices other than water, soil, sludges and oils.

Matrix Spikes/Matrix Spike Duplicates are similar to the Laboratory Control Sample except the analytes used for spiking are added to a second and third separate aliquot from the same container of a selected few client samples in a batch of analyses. When prepared and analyzed, MS/MSD's can also provide a measure of overall precision. They incorporate sample matrix effects and field conditions. MS/MSD's are routinely prepared at a frequency of 10% when adequate sample volume is provided.

Surrogates provide an estimate of bias based on recovery of similar compounds, but not the compounds analyzed, for each sample, incorporating sample matrix effects and field conditions. Surrogates are added to all samples analyzed by GC/MS and certain GC analyses prior to sample preparation.

An Internal standard is an analyte that has the same characteristics as the surrogate, but is added to each sample in a batch, just prior to analysis and is used for quantitation. It corrects for bias or change in instrument performance from sample to sample, incorporating matrix effects associated with the analysis process only.

Precision Measurements

A Laboratory duplicate is a laboratory control sample that has been homogenized and split into two equal portions before the method sample preparation process. It measures method precision associated with the preparation through analysis and is prepared and analyzed at a rate of one per batch or one per twenty samples whichever is greater.

A **Sample duplicate** is a sample that has been homogenized and split into two equal portions before the method sample preparation process. It measures sample precision associated with the preparation through analysis and is prepared and analyzed at a rate of one per batch or one per twenty samples whichever is greater in the inorganic laboratories. For organic analyses the MS/MSD fills this function.

11.2 Control Charts

Control charts are quality control tools which graphically display the QC parameters over time. Both accuracy (Figure 11-1) and precision (Figure 11-2) control charts are maintained for each method and matrix. Each chart can be broken into three parts: sample identification, sample response/calculation, and graphic representation (the plot).

11.2.1 Accuracy

For certain analytical programs (NEESA, AFCEE), accuracy charts are maintained for Surrogate and Laboratory Control Sample recovery. Each sample is identified by the date it was analyzed and its PACE NE sample number. The true value (three-significant-figure expected value based on the amount spiked) is denoted as SR. This amount is fixed for each method and matrix to be approximately five to ten times the MDL. The experimental value (three-significant-figure observed value) is denoted by SA and varies for each sample based on the recovery of the amount spiked. From these two values the percent recovery (%R) is calculated as:

$$\%R = (SR / SA) * 100$$

except for the matrix spike sample where the concentration present in the original sample aliquot must also be factored in. For matrix spike samples, the percent recovery is calculated as:

$$(SSR-SR)/SA \times 100$$

where: SSR is the spiked sample recovery
SR is the original sample recovery
SA is the amount of spike added

The percent recovery is plotted onto the graph where:

- the x-axis is the number of data points per page; and
- the y-axis is the range of percent recover .

11.2.2 Precision

Precision charts are maintained for LCS duplicates. Both sa identified by the date(s) analyzed and their PACE NE number. s the three-significant-figure observed value for ' rig sample; D denotes the three-significant-figure observed value for t uplicate. The comparison of the two values is expressed as relative percent difference (RPD), where relative percent difference is calculated to be an absolute value of three significant figures greater or equal to zero. This calculation is as follows:

$$RPD = \frac{S-D}{[(S+D)/2]} \times 100$$

The relative percent difference is plotted on the graph whe

- the median, zero, represents 0% difference
- the x-axis is the number of data points per chart; and
- the y-axis is the range of relative percent differences.

11.2.3 Limits

Both upper and lower warning limits and upper and lower control limits are established to aid in interpreting a suspicious or an out-of-control event. Warning limits express a narrower confidence interval and are use warn the analyst or supervisor of possible system inconsistencies efore an out-of-control event occurs. Control limits express th of accepted method variability. Control limits and warning nits are updated quarterly.

Warning Limits

When not mandated by the method, PACE NE adopt warning limits e the mean ± 2 standard deviations or a 95% confidence interval, where:

$$\text{Mean} \quad \bar{x} = \frac{1}{n} \sum_{i=1}^{n} x_i$$

$$\text{Standard Deviation} \quad s^2 = \frac{\sum_{i=1}^{n} x_i^2 - (1/n)\left(\sum_{i=1}^{n} x_i \right)^2}{n-1}$$

where: $\bar{X}$ = mean value
X = individual values
n = total number of values

Control Limits

Unless otherwise specified by the analytical method in use or by the project QA plan, PACE NE uses the 99% confidence interval as the control limits, which is defined as the mean ± 3 standard deviations. Where interlaboratory expected ranges have been determined, PACE NE's goal is for their control limits to fall within these multi-laboratory expected ranges for that method.

Both warning and control limits are determined and published at the end of each quarter for each method and matrix based only on that quarter's performance. However, a running mean and standard deviation is also calculated with each addition of 20 new data points and is available to those interested.

Suspicious/Out-of-Control Events

Plotting and connecting successive data points on control charts enables the laboratory to detect many types of suspicious and out-of-control situations. These events can be caught by monitoring the following: outliers (suspicious and out-of-control), runs (suspicious), trends (suspicious), and periodicity (suspicious).

Outliers

There are two types of outliers: any particular point that falls outside the control limits or any point that falls outside the warning limits. A point that falls outside the control limits is classified as an out-of-control event; a point that falls outside the warning limits is classified as a suspicious event.

Runs

A run is defined as a series of points that line up on one side of the central line (the mean). Any run that has a length of seven points is indicative of a potential abnormality in the process, a suspicious event. A run can suggest several potential problems such as a leak in the system, elevated contamination, or incorrect dilutions of standards.

Trends

A trend is defined as a series of points that are marked by an unbroken rise or fall. Any trend with a length of five points is classified as a suspicious event. A trend may indicate a change in instrument sensitivity due to a dirty source or injection port or standard degradation, to name a few.

Periodicity

Periodicity is a term used to describe a recurring pattern of change over equal intervals. This occurrence may be of any length or amplitude; thus, careful observation of the control chart is necessary.

11.3 Utilization of Quality Control Data

The purpose for preparing and analyzing quality control samples is to demonstrate, through the known entities, how accurate and precise the investigative sample data are. Tables 11-1 and 11-2 summarize the quality control assessment criteria by matrix for the most commonly used methods by this laboratory. Different criteria may be dictated by different methods or by project QA plans.

TABLE 11-1
TYPICAL QUALITY CONTROL CRITERIA FOR ORGANIC ANALYSES

Parameter	Type	Frequency	Compounds	Performance Criteria	
WATER					
Volatiles (method 8240)	Method blank Surrogate spike	1/20 or 1/batch every sample	— $D_4$1,2-dichloroethane BFB D_8-toluene	± D.L. 70 to 138% 72 to 132% 86 to 114%	
				Recovery	RPD
	Laboratory Control Sample, MS/MSD	1/20 or 1/batch	Benzene 1,1-dichloroethene Trichloroethene Chlorobenzene Toluene	76 to 127% 61 to 145% 71 to 120% 75 to 130% 71 to 125%	11 14 14 13 13
	Calibration check	every 12 hours		≤ 25% difference for CCC compounds; minimum response factor for SPCC compounds	
Base/neutral Acid compounds (method 8270)	Method blank Surrogate spike	1/20 or 1/batch every sample	— D_5 -phenol 2-fluorophenol 2,4,6-tribromophenol D_5-nitrobenzene 2-fluorobiphenyl D_{14}-terphenyl	± D.L. 10 to 110% 21 to 110% 10 to 123% 35 to 114% 43 to 116% 33 to 141%	
				Recovery	RPD
	Laboratory Control Sample, MS/MSD	1/20 or 1/batch	Phenol 2-chlorophenol 1,4-dichlorobenzene N-nitrosodipropylamine 1,2,4-trichlorobenzene 4-chloro-3-methylphenol Acenaphthene 2,4-dinitrotoluene 4-nitrophenol Pentachlorophenol Pyrene	12 to 89% 27 to 123% 36 to 97% 41 to 116% 39 to 98% 23 to 97% 46 to 118% 24 to 96% 10 to 80% 9 to 103% 26 to 127%	42 40 28 38 28 42 31 38 50 50 31
	Calibration check	every 12 hours		≤ 25% difference	
Pesticides & PCBs (method 8080)	Method blank Surrogate spike	1/20 or 1/batch every sample	— Dibutyl chlorendate	± D.L. 24 to 154%	
				Recovery	RPD
	Laboratory Control Sample, MS/MSD	1/20 or 1/batch	Lindane Heptachlor Aldrin Dieldrin Endrin 4,4-DDT	36 to 123% 40 to 131% 40 to 120% 52 to 126% 56 to 121% 38 to 127%	15 20 22 18 21 27
	Calibration check	every 10 samples		≤ 15% difference	

TABLE 11-1 (Continued)

Parameter	Type	Frequency	Compounds	Performance Criteria	
SOIL					
Volatiles (method 8240)	Method blank	1/20 or 1/batch	—	± D.L.	
	Surrogate spike recovery	every sample	$D_4$1,2-dichloroethane	70 to 138%	
			BFB	72 to 132%	
			D_8-toluene	86 to 114%	
				Recovery	RPD
	Laboratory Control Sample, MS/MSD.	1/20 or 1/batch	Benzene	66 to 142%	21
			1,1-dichloroethene	59 to 172%	22
			Trichloroethene	62 to 137%	24
			Chlorobenzene	60 to 133%	21
			Toluene	59 to 139%	21
	Calibration check	every 12 hours		≤ 25% difference for CCC compounds; minimum response factor for SPCC compounds	
Base/Neutral Acid compounds (method 8270)	Method blank	1/20 or 1/batch	—	± D.L.	
	Surrogate spike	every sample	D_5 -phenol	24 to 113%	
			2-fluorophenol	25 to 121%	
			2,4,6-tribromophenol	19 to 122%	
			D_5-nitrobenzene	23 to 120%	
			2-fluorobiphenyl	30 to 115%	
			D_{14}-terphenyl	18 to 137%	
				Recovery	RPD
	Laboratory Control Sample, MS/MSD	1/20 or 1/batch	Phenol	26 to 90%	35
			2-chlorophenol	25 to 102%	50
			1,4-dichlorobenzene	28 to 104%	27
			N-nitrosodipropylamine	41 to 126%	38
			1,2,4-trichlorobenzene	38 to 107%	23
			4-chloro-3-methylphenol	26 to 103%	33
			Acenaphthene	31 to 137%	19
			2,4-dinitrotoluene	28 to 89%	47
			4-nitrophenol	11 to 114%	50
			Pentachlorophenol	17 to 109%	47
			Pyrene	35 to 142%	36
	Calibration check	every 12 hours		≤25% difference	
Pesticides & PCBs (method 8080)	Method blank	1/20 or 1/batch	—	± D.L.	
	Surrogate spike	every sample	Dibutyl chlorendate	18 to 150%	
				Recovery	RPD
	Laboratory Control Sample, MS/MSD	1/20 or 1/batch	Lindane	46 to 127%	50
			Heptachlor	35 to 130%	31
			Aldrin	34 to 132%	43
			Dieldrin	31 to 134%	38
			Endrin	42 to 139%	45
			4,4-DDT	23 to 134%	50
	Calibration check	every 10 samples		≤ 15% difference	

TABLE 11-2
TYPICAL QUALITY CONTROL CRITERIA FOR INORGANICS ANALYSES

Soil and Water

Parameter	Type	Frequency	Performance Criteria
Metals by ICP (Method 6010)	Calibration Blank	every calibration	± D.L.
	Calibration verification	every calibration	90 to 110%
	Continuing calibration	1/10 samples	90 to 110%
	Method blank	1/20 or 1/batch	± D.L.
	Laboratory Control Sample/LSC dup	1/20 or 1/batch	80 to 120%
	Sample duplicate	1/20 or 1/batch	± D.L. or 20% RPD
	Matrix Spike	1/20 or 1/batch	75 to 125%
Metals by Furnace AA (7000 series methods)	Calibration blank	every calibration	± D.L.
	Calibration verification	every calibration	90 to 110%
	Continuing calibration	1/10 samples	90 to 110%
	Method blank	1/20 or 1/batch	± D.L.
	Laboratory Control Sample/LCS dup	1/20 or 1/batch	80 to 120%
	Sample duplicate	1/20 or 1/batch	± D.L. or 20% RPD
	Matrix Spike	1/20 or 1/batch	75 to 125%
	Duplicate injections	every sample and standard	± 20% RPD
Cold Vapor AA (Mercury)	Calibration blank	every calibration	± D.L.
	Calibration verification	every calibration	80 to 120%
	Continuing calibration	1/10 samples	80 to 120%
	Method blank	1/20 or 1/batch	± D.L.
	Lab control sample/LCS Dup	1/20 or 1/batch	80 to 120%
	Sample duplicate	1/20 or 1/batch	± D.L. or 20% RPD
	Matrix Spike	1/20 or 1/batch	75 to 125%
Total Petroleum Hydrocarbons	Calibration blank	every calibration	± D.L.
	Calibration verification	every calibration	85 to 115%
	Continuing calibration	1/10 samples	85 to 115%
	Method blank	1/20 or 1/batch	± D.L.
	Lab control sample	1/20 or 1/batch	75 to 125%
	Sample duplicate	1/20 or 1/batch	± 30% RPD
Total Cyanide	Calibration blank	every calibration	± D.L.
	Calibration verification	every calibration	85 to 115%
	Continuing calibration	1/10 samples	85 to 115%
	Method blank	1/20 or 1/batch	± D.L.
	Laboratory Control Sample/LCS dup	1/20 or 1/batch	80 to 120%
	Sample duplicate	1/20 or 1/batch	± 30% RPD
	Matrix Spike	1/20 or 1/batch	75 to 125%
General Wet Chemistries	Calibration blank	every calibration	± D.L.
	Calibration verification	every calibration	85 to 115%
	Continuing calibration	1/10 samples	85 to 115%
	Method blank	1/20 or 1/batch	± D.L.
	Laboratory Control Sample/LCS dup	1/20 or 1/batch	80 to 120%
	Sample duplicate	1/20 or 1/batch	± 30% RPD
	Matrix Spike	1/20 ro 1/batch	75 to 125%

Figure 11-1
Examples of Accuracy Control Chart

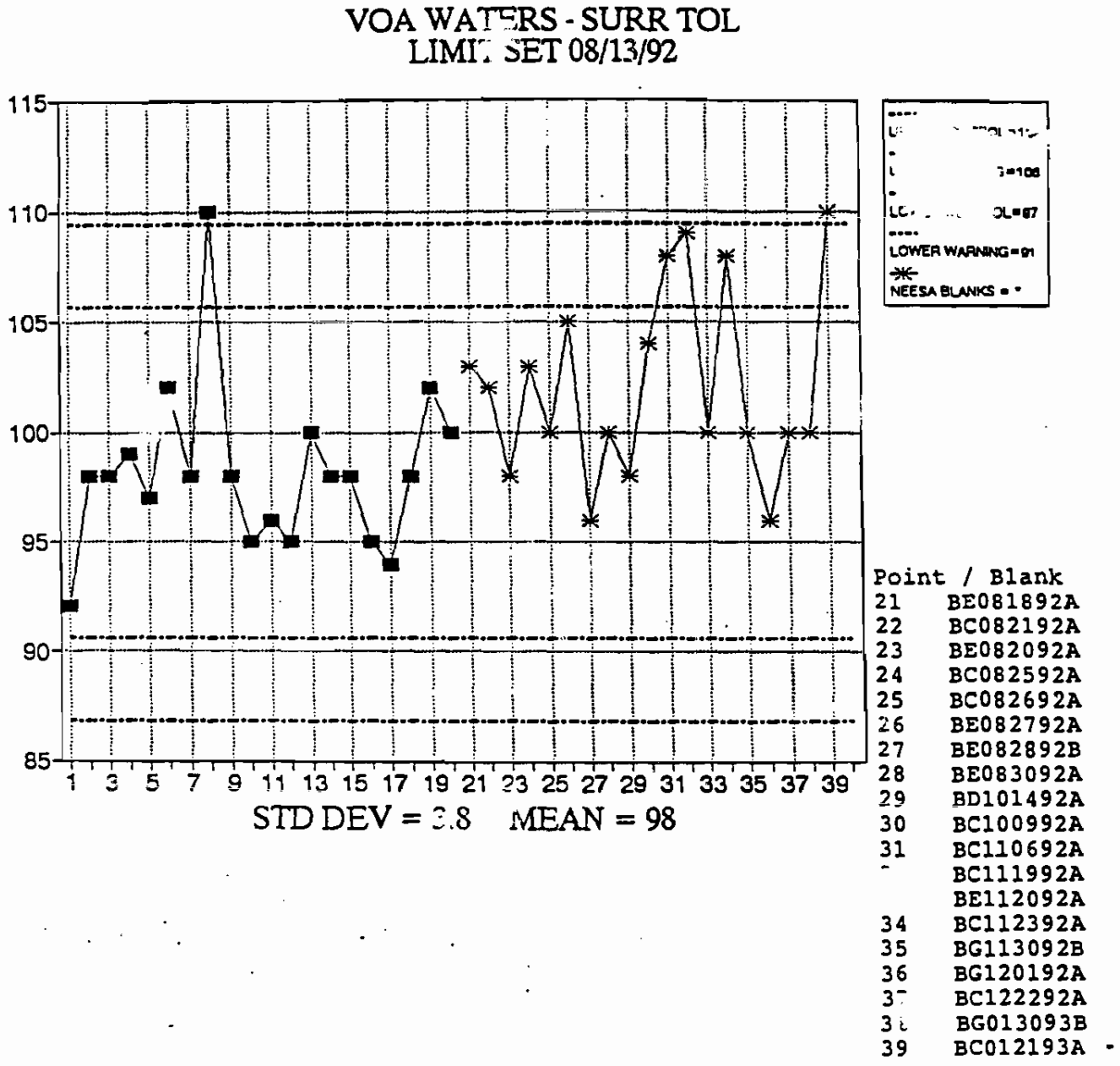
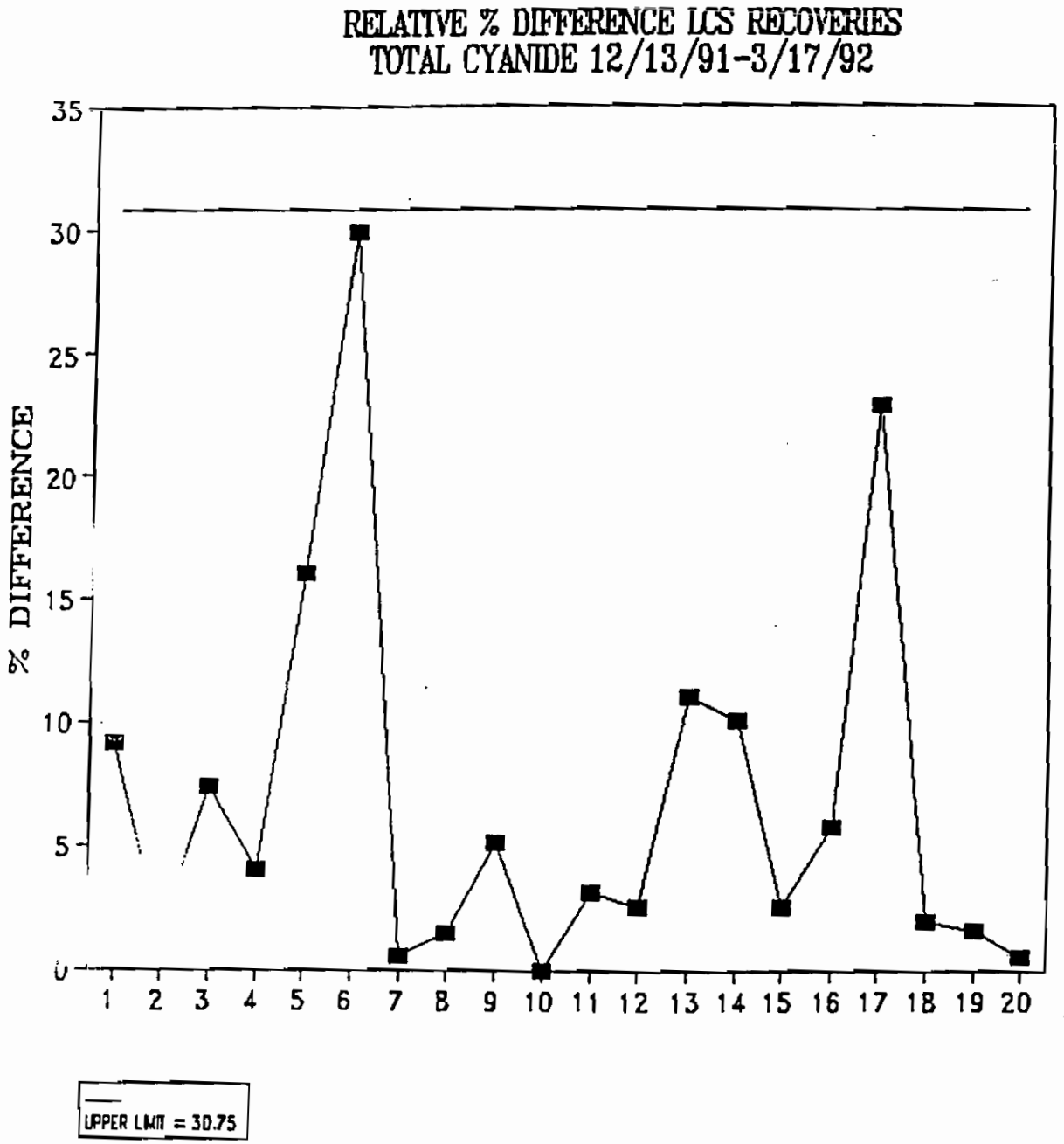


Figure 11-2
Examples of Precision Control Chart



12.0 STANDARD OPERATING PROCEDURES

12.1 Purpose and General Provisions

Standard Operating Procedures (SOPs) are formal, revision-controlled documents that:

1) define, to PACE NE's clients and to regulatory agencies, the methods used by PACE NE in the performance of tasks having an effect on the quality of data, findings or conclusions;

2) establish the basis for similar training of personnel and set a standard for assessment;

3) provide standard methods for execution and documentation of work, to maximize consistency, uniformity and reliability of products; and

4) facilitate coordination among individuals performing separate, but interdependent tasks.

SOPs describe standard methodologies that may at times be inappropriate for a specific project. In such cases, exceptions to the SOPs are stated in an SOP Deviation Form, with rationale. The SOP Deviation form is kept as part of the data package archived at PACE NE.

12.2 Responsibilities

The Laboratory Section Managers are responsible for determining, through consultation with the Quality Assurance Officer and PACE NE Management, the activities that require SOPs, and for working with the appropriate technical personnel to develop the SOPs.

The Quality Assurance Department is responsible for obtaining technical review and approval of SOPs, for maintaining control of new SOPs and revisions, and for maintaining an up-to-date distribution list for SOPs.

PACE NE personnel are responsible for performing tasks in accordance with applicable SOPs, except as explicitly directed by the relevant Quality Assurance Project Plan, contract, or Health and Safety policy. PACE NE personnel are also responsible for assisting in designing accurate and practical SOPs and in keeping the SOPs up-to-date.

Technical reviewers of SOPs are responsible for providing review of drafts sent to them within the schedule indicated in the request.

12.3 Minimum Contents of SOPs

Each Standard Operating Procedure shall contain at a minimum, the following information:

Title - The name of the concerned task

SOP Number - The internal document control number assigned and tracked by the QA Department

Acceptance - The signature of the originator(s), Quality Assurance Officer and appropriate operations management authority to officially adopt the procedure

Date - date of issue of most recent revision

Section 1.0 - Purpose and Applicability - An explanation of the objectives of the procedure, typical applications and limitations

Section 2.0 - Definitions - A listing of any terms, expressions, or acronyms found in the procedure

Section 3.0 - Applicable Documents/References - A listing of pertinent, supporting procedure or reference documents such as methods, manuals and/or SOPs

Section 4.0 - Apparatus and Materials - A complete list of the equipment, apparatus, reagents, etc. needed for the procedure

Section 5.0 - Method/Procedure - A clear description of the task on a step-by-step basis. The method description should be written clearly enough, and in sufficient detail, to ensure that any two persons performing the procedure will achieve equivalent results, and to provide clients and reviewing agencies with a thorough understanding of the procedure. Acceptable and equivalent alternatives should be addressed whenever possible, and described in the same detail

Section 6.0 - Quality Control Requirements and Acceptance Criteria - An outline of quality control requirements, including procedures frequency requirements and acceptance criteria. Acceptance criteria may take the form of an illustration such as a

chart of acceptable results with tolerances, or other appropriate forms

Section 7.0 - Calculations, Data Management and Reporting of Results - A summary of the automated and manual calculations performed as well as reporting requirements, including data flow charts as appropriate

Section 8.0 Corrective Actions - A description of what must be done, when and by whom in instances when the QC requirements are not met

Section 9.0 Responsibilities - Identification of the individuals (by title or organizational position) who are responsible for performing and facilitating the tasks governed by the SOP

Section 10.0 Health and Safety Considerations - A discussion of specific Health and Safety issues that must be considered prior to and during the performance of the procedure described.

12.4 SOP Development and Approval

Laboratory SOPs are developed by the laboratory's technical staff, working with the QA Department (PACE NE SOP QA-553 Preparation of SOP's). The QA Department will also assist by assigning SOP numbers and by coordinating word processing, review and approval. Laboratory SOPs must be reviewed and approved by the management of the laboratory operations to which the SOPs apply. Thus, the following people must review and approve each laboratory SOP:

- the Supervisor of the specific operation to which the SOP pertains if applicable) or the Laboratory Manager - This signature indicates that the written SOP reflects the current practice in the laboratory.
- the Laboratory Technical Director - This signature indicates that the SOP is technically adequate to handle the analysis of environmental samples expected to be received at PACE NE and technically compliant within the framework given in Section 8.0 and with any known exceptions noted.
- the Quality Assurance Officer - This signature indicates that the SOP has been reviewed for compliance with the referenced methods and that desrepencies between the method and practice have been resolved.
- the PACE NE Regional Director - This signature signifies a management review and approval of the practices detailed in the SOP.

12.5 Numbering

Each SOP is assigned a unique PACE NE number from the Inventory of PACE NE SOPs, maintained by the QA Department. Each laboratory section has been assigned a block of numbers.

12.6 Revisions

SOP revisions may be necessitated by regulatory requirements, technological advancements or other causes, but not by the requirements of a single project alone. Contradictions between standard procedures and the requirements of a specific project are resolved in the quality assurance plan for that project and are controlled internally through the generation of an SOP Deviation Form (Figure 12-1).

Revisions may be proposed initially by the Quality Assurance Department or they may be recommended by users. Recommendations for revisions must be sent to the Quality Assurance Department.

Technical changes to an SOP need to be made immediately, but revisions must not be made by an individual to only his/her personal copy. If there are changes to an SOP, they need the approval of the Group Supervisor, Lab Manager and QA and must be made manually to ALL copies of that SOP in use in the lab. A formal revision should be initiated ASAP. Recommendations for minor revisions will be accumulated by QA until sufficient to warrant a document revision.

Revisions are accomplished by the preparation of a new typed draft with the changes incorporated and listed on the cover page. (The cover page is a permanent document and stays with the SOP despite revisions.) Approval of the revisions is signified by dated acceptance signatures adjacent to the listed revisions in the lower section of the cover page. The QA Officer is authorized to approve minor revisions. Revisions which effect the technical approach or content will also require review and approval of a technical or section manager. Once formally accepted, the revised document replaces the previous version and is distributed to controlled copy holders with instructions as to what document(s) it replaces.

Occasionally, revisions are significant enough to warrant a complete rewrite. In such cases, the changes are not listed on the cover page. Instead the words "complete rewrite" are entered and the new document must undergo review and approval as for a new SOP. The judgement as to whether a complete rewrite is required shall be made by the Quality Assurance Officer.

Technical revisions and complete rewrites will necessitate training recertification for all personnel involved. The laboratory or section manager is responsible for ensuring that training is accomplished and documented. Required training documentation and instructions will be distributed with the SOPs by the QA Department.

12.7 Distribution

The QA Department distributes SOPs to technical staff and maintains distribution lists to ensure that revisions and new SOPs are distributed to all responsible individuals. The QA Department maintains a complete set of up-to-date SOPs and distributes them as required. An SOP archive is maintained by QA Department and SOPs are distributed from that archive.

12.8 SOP Archive

An archive of all PACE NE SOPs, in the form of both hard-copy and electronic masters of current revisions, are maintained by the Quality Assurance Department. The hard-copy archive also contains a hard-copy master of all obsolete versions of each revised SOP.

Access to originals is obtained through QA personnel.

Figure 12-1
SOP Deviation Form
(Revision 1, 1/93)

SOP Number and Title:_____

Date and Signature_____

Lab Number/Job/or Client deviation applies to:

_____
_____
_____

Describe deviation in detail, including normal procedure and reason for deviation:

_____
_____
_____
_____
_____
_____
_____
_____
_____
_____

Approval/Date

_____Date_____
Technical Director

_____Date_____
Quality Assurance Officer

_____Date_____
Regional Director

13.0 PERFORMANCE AND SYSTEM AUDITS

PACE NE's Analytical Chemistry Laboratory participates in a variety of interlaboratory tests and intralaboratory and performance checks to provide periodic assessment of the effectiveness of the overall quality control program.

13.1 Interlaboratory Performance Surveys

Performance surveys conducted by the EPA and the New Hampshire Department of Environmental Services constitute the bulk of interlaboratory comparisons.

- EPA Performance Evaluations - Water Supply - Semiannual (April and September)
  - Trace Metals
  - Nitrate/Nitrite/Fluoride
  - Insecticides
  - Herbicides
  - PAH's
  - Adipate/Phthalates
  - Trihalomethanes (THMs)
  - Volatile Organic Compounds
  - Residual Free Chlorine
  - Turbidity
  - Total Filterable Residue
  - Calcium (as $CaCO_3$)
  - pH
  - Alkalinity
  - Corrosivity
  - Sodium
  - Sulfate
  - Total Cyanide
- EPA Performance Evaluations - Water Pollution - Semiannual (February and August)
  - Trace Metals
  - Minerals
  - Nutrients

- Demand
- PCB's
- PCB's in Oil
- Pesticides
- Volatile Halocarbons
- Volatile Aromatics
- Total Cyanide
- Non-Filterable Residue
- Oil and Grease
- Total Phenolics

PACE NE's performance is evaluated by the respective agency after each round of testing, and reported to the Laboratory Technical Director. The Laboratory Technical Director forwards the results to the QA Auditor in charge of certifications. The QA Auditor distributes copies of the results to section managers and the QA Officer.

In addition to the EPA WP/WS performance evaluation studies, PACE NE participates in a number of different state, federal and commercial studies such as the State of New York, US Navy NEESA, US Army COE, USEPA CLP Quarterly Blind samples, US EPA DMR PE studies, and Chemical Waste Management round robin studies.

13.2 Periodic Internal Audits

Internal auditing is conducted by the QA Auditors. These audits occur at least every month, and typically focus on either performance relative to an SOP or a specific project. Internal audits take two forms - performance audits and systems audits. Performance audits involve submittal of blind spikes to the laboratory by the Quality Assurance Department for assessment of analytical accuracy. Systems audits consist of a thorough review of procedures and documentation to confirm that work was performed in accordance with this Manual, SOPs, and/or project QA Plan and that adequate documentation exists to satisfy the project requirements.

13.2.1 Performance Audits

Audit Standards

As required on specific projects, the Q ity Assurance Department provides spikes for analysis as independent chec amples (audit standards). The QA Department prepares any audit standards that can be prepared readily from relatively non-hazardous, pure materials or certified concentrated standards. In

some cases, preparation of reliable audit standards requires special facilities and equipment due to the hazardous nature of the materials and/or the requirement for precise measurement of minute quantities. In such cases, audit standards are obtained from the USEPA, Environmental Monitoring and Support Laboratory (EMSL), Cincinnati, Ohio, or from an equivalent source. The nature of the audit standards and the frequency of performance audits are specified in the Quality Assurance Plan of each project for which performance auditing is required. When practical, audit standards are provided in matrices resembling real project sample matrices, and undergo the full sample preparation and analysis procedure. However in many cases this is impractical, and it is necessary to submit audit samples as extracts, for analysis only. All measurable constituents in the audit standards should be within the expected range of concentrations to be encountered in the real samples (or in the extracts).

13.2.2 Systems Audits

There are two different types of laboratory systems audits. Systems audits of laboratory operations are performed at a minimum frequency of once every month. Systems audits address general laboratory operations and conformance to the Laboratory Quality Assurance Manual. Some project quality assurance plans require project-specific laboratory systems audits (Project Audits).

Systems Audit Procedures

The systems audits are performed by the Quality Assurance Auditors. Audit checklists are used to ensure that all salient points are addressed and documented. The checklists are filled out legibly and reproduceably, in ink, by the auditor, and are signed and dated by the auditor when completed. The audit checklist is based on EPA laboratory evaluation criteria, the provisions of the Laboratory Quality Assurance Manual and PACE NE SOPs. Project audit checklists are drawn from the applicable QAPPs, as well as relevant provisions of the QA Manual.

Audit checklists will cover at least the following areas:

- Systems Audit
    - Personnel qualifications and training records
    - Adequacy of laboratory facilities, including work space, lighting, ventilation, and supplies
    - Maintenance and calibration recordkeeping for analytical

equipment
- Safety (facility configuration and practices)
- General operations, including glassware cleaning, inventory and checking of reagents and standards, and storage procedures
- Recordkeeping, including sample log-in and tracking, traceability of standards, control charts, and raw data recording and tracking.

- Project Audit

  - Sample log-in and chain-of-custody records
  - Sample storage procedures and records
  - Sample preparation and analysis procedures
  - Method validation (where applicable)
  - Conformance to QAPP
  - Control charts
  - Precision and accuracy assessment
  - Method blanks, reagent blanks, duplicates, check samples, fortifications, surrogates, etc.
  - Calibration
  - Data packages
  - Analyst qualifications
  - Data validation and reporting

13.3 QA Reporting and Corrective Action

Each systems audit is immediately followed by a debriefing, in which the auditor discusses his/her findings with the laboratory representatives. The debriefing serves a two-fold purpose. First, laboratory management is afforded an early summary of findings, which allows them to begin formulating corrective strategies, and second, the auditor has a chance to test preliminary conclusions and to correct any misconceptions before drafting his report.

The systems audit report (which may or may not contain performance audit findings) is issued to the Laboratory Manager and appropriate supervisors and personnel for corrective action. These responses are forwarded, in writing, to the auditor. The auditor then circulates the report to the QA Officer, laboratory management and to company management.

Results of interlaboratory performance surveys and in-house audits, along with unresolved corrective action items are summarized in a monthly report from the Quality Assurance Officer to the PACE NE Regional Director.

14.0 PREVENTIVE MAINTENANCE

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument. Designated laboratory personnel are trained in routine maintenance procedures for all major instrumentation. When repairs are necessary, they are performed by either trained staff or instrument manufacturer service personnel.

SOPs are written for each instrument that cover basic operation and maintenance procedures (QA-800, Use, Calibration, and Maintenance of Equipment - Inorganics Lab, QA-801, Use, Calibration, and Maintenance of Equipment - Organics Lab). Detailed logbooks documenting preventive maintenance, non-routine maintenance and repairs are also maintained for each instrument. The following are brief summaries of maintenance for each major instrument.

14.1 Preventive Maintenance - GC/MS

Regularly performed maintenance includes, but is not limited to the following for GC/MS instrumentation:

- hard tune with calibration gas (PFTBA)
- removal of 2-3 inches from the injection end of the capillary columns
- replacement of 2-3 inches of column packing from the injection end of packed columns
- injection port liner replacement
- replace injection port septum
- clean ion source as needed
- check vacuum pump oil level
- check carrier gas tanks
- replace or recondition vent traps

14.2 Preventive Maintenance - GC

Regularly performed maintenance includes, but is not limited to the following for GC instrumentation:

- replacement of 2-3 inches from the injection end of the capillary columns
- removal of 2-3 inches of column packing from the injection end of packed columns
- injection port liner replacement
- replace septum
- check carrier and support gases
- NRC wipe test ECD

14.3 Preventive Maintenance - ICP

- check liquid argon tank level
- change pump tubing
- clean nebulizer and spray chamber as needed
- replace and realign plasma torch when required
- check cooling system water level
- empty waste reservoir when full

14.4 Preventive Maintenance - AA Graphite Furnace

- check and align source lamps
- clean and inspect graphite tube, replacing when surface appears excessively burnt or cracked
- clean and inspect contact ring, replacing when excessively worn

- clean mirrors for optical sensor and sample compartment windows
- check autosampler injector alignment and deposition

14.5 Preventive Maintenance - Mercury Analyzer

- Check and align source lamp
- remove and clean sample cell and connecting tubes
- check sparger for proper operation
- clean sample compartment windows

14.6 Preventive Maintenance - General Laboratory Areas

- clean and calibrate balances biannually
- check balance calibration each day of use
- clean balance pan prior to each use
- calibrate class "S" weights every two years
- calibrate automatic pipets and burets monthly
- calibrate thermometers yearly against an NBS traceable thermometer
- record refrigerator, freezer, and oven temperatures each weekday
- clean, check, calibrate to manufacturers specifications all pH, DO, conductivity and, turbidity meters, and spectrophotometers biannually
- general housekeeping: keep counter tops, hoods, and floors clean
- check airflow in hoods once a week

15.0 CORRECTIVE ACTION

When errors, deficiencies, unusual occurrences, or out-of control situations exist, the QA program provides systematic procedures, called "corrective actions", to resolve problems and restore proper functioning to the analytical system. Within PACE NE, a distinction is made between "out-of-control events" and "unusual occurrences" for the purposes of requiring corrective actions.

An out-of-control event is any event which is beyond the acceptance limits established for laboratory operation by PACE NE SOP's, EPA methods, or client specific contracts or protocols. This can be due to data which are outside of the accepted bounds for accuracy and/or precision, method contamination, improper instrument calibration or maintenance, or deviations from the contract or SOP detected by a QA audit.

An unusual occurrence is a situation in which the analytical system is, strictly speaking, compliant with the protocol or SOP and therefore in control but an atypical or undesirable incident has occurred which warrants further investigation. Such an occurrence could be a holding blank which is contaminated or differences in the pattern of non-spiked target compounds between a spiked and unspiked aliquot of a sample used as the matrix spike.

Both out-of-control events and unusual occurrences are noted on a PACE NE Corrective Action Report (Figure 15-1). A corrective action report must be generated whenever either type of event is noted.

15.1 Out-of Control Events

Out-of-control events associated with the statistical analysis and review of data are straight forward to identify. The analyst generating the data is responsible for checking the results against the established limits. Any deviations are immediately addressed. If data are outside accepted limits, the analyst immediately notifies the responsible section supervisor. If the situation is not corrected so that an out-of-control condition occurs, or is expected to, the section supervisor shall notify the Laboratory Manager and the Quality Assurance Officer. The Laboratory Manager and Section Supervisors are responsible for identifying the source of the problem and initiating corrective action. Completion of corrective action should be evidenced by the return of data to prescribed acceptable limits.

Events which do not readily cause an immediate obvious effect on data quality are more difficult to identify. Such events could be samples stored at an incorrect temperature or held beyond prescribed holding times, or improper maintenance of records. Everyone in the laboratory is

responsible for reporting "system" problems. Analysts should report out-of-control events to their supervisor, the Section Supervisors, who should in turn report to the Laboratory Manager. Corrective action is again the responsibility of the Laboratory Manager and the Section Supervisors. They shall review and approve the action taken.

If an out-of-control event does occur during analysis, for instance an LCS recovery falls outside the expected range, the analyst must describe on the corrective action report the event, the investigative and corrective actions taken, the cause of the event, and notify the QA Officer. In some cases, investigation of an out-of-control event will reveal no problems. In such cases, only the event and the investigative action is recorded.

The investigative action taken is somewhat dependent on the analysis and the event. However, listed below is a progression of steps which may be taken to find the cause of an out-of-control event:

- Check calculations to ensure there are no errors
- Check standard and spiking solutions for degradation or contamination
- Check instrument performance

If the problem is with the standards or instrument performance, the analyst must recalibrate or retune the instrument before reanalyzing the sample extracts affected. If the out-of-control condition is still not remediated, the samples may require reextraction and reanalysis or data qualification.

It is occasionally necessary to qualify data when the accompanying quality control data are not within established performance criteria. The qualifying of data alert the data end user to the fact that the analysis was, to some degree, flawed and that the precision and accuracy of the data produced may not fulfill the data quality objectives (DQOs) for that particular project. Based on the project DQOs, analytical data with qualifiers may not be appropriate for the intended use.

15.1.1 Volatile Organic Analyses

Method Blanks

If target compounds are detected in the method blank above the detection limit (above 5 times the detection limit for methylene chloride, acetone, toluene, and 2-butanone) the corrective action consists of checking the calculations, reanalyzing the blank, qualifying the associated sample data, and investigating the source of the problem to implement corrective action for the future. When target list compounds are detected in a method blank, the following condition applies:

- When any target compound is detected in a method blank above the action levels listed earlier, but not in associated samples, then no qualifier is applied.

Surrogates

The % recovery of the surrogates is calculated for each sample, blank, and LCS. Corrective action is taken whenever one (or more) surrogate recovery is outside the acceptance criteria. The following corrective actions are taken when required as stated above:

- Check calculations to assure there are no errors;
- Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;
- If instrument failure is indicated, reanalyze the sample;
- If a method blank surrogate is outside of acceptance criteria, then the problem must be corrected before proceeding with sample analysis. This may include reanalysis, reextraction or recalibration;
- If the surrogate could not be measured because the sample required a dilution, no corrective action is required. The recovery of the surrogate is recorded with the note "surrogate diluted out".
- If all QC associated with the sample is within acceptance limits (the method blank surrogate recovery and LCS spike recovery), the problem may be attributed to a matrix effect. Samples exhibiting a matrix effect will be qualified and discussed in the report narrative.

Laboratory Control Sample/Laboratory Control Sample Duplicates

The % recovery of the Laboratory Control Samples (LCS) and the relative percent difference (RPD) for LCS Duplicates are calculated. Corrective action is taken whenever one (or more) recovery or RPD is outside the established acceptance criteria. The following corrective actions are taken when required as stated above:

- Check calculations to assure there are no errors;

- Check internal standard and spiking standard solutions for degradation, contamination, etc., and check instrument performance;

- Reanalyze samples associated with a failed LCS, if available;

- If that does not correct the problem, then the data is reported and a qualifying statement included in the report narrative.

For Matrix Spike and Matrix Spike Duplicates, if all QC associated with a sample is within acceptance limits (the method blank and LCS/LCS dup spike recovery), the problem may be attributed to a matrix effect. Samples exhibiting a matrix effect will be qualified and discussed in the report narrative.

Calibration

For an initial 5 point calibration curve to be valid, the % relative standard deviation of the individual relative response factors (RRF) for the Calibration Check Compounds (CCC) shall be less than or equal to 25%. If this criteria is not met, then the calibration curve shall be reanalyzed.

For continuing calibration checks to be valid, the relative response factor for each of the System Performance Check Compounds (SPCC) should be at least 0.300 (0.250 for Bromoform) and the RRF for each of the CCC should be ≤25% different from the average RRF from the initial calibration. If these criteria are not met, then the following corrective actions should be taken:

- Check internal standard and standard solutions for degradation, contamination, etc.,

- Check instrument for contamination at the injection port inlet and front end of the column;

- If no source of the problem is identified, then a complete 5 point initial calibration must be performed.

The SPCC and CCC for Volatiles are:

SPCC	CCC
Chloromethane	Vinyl Chloride
1,1-Dichloroetnane	1,1-Dichloroethene
Bromoform	Chloroform

1,1,2,2-Tetrachloroethane	1,2-Dichloropropane
Chlorobenzene	Toluene
	Ethylbenzene

15.1.2 Semivolatile Organic Compounds

Method Blanks

If target compounds are detected in the method blank above the detection limit (above 5 times the detection limit for phthalate esters) the corrective action consists of the following:

- checking the calculations;
- reanalyzing the blank;
- flagging the associated sample data;
- investigating the source of the problem to implement corrective action for the future.

When target list compounds are detected in a method blank, the following data condition applies:

- when any target compound is detected in a method blank above the action levels listed earlier but not in associated samples, then no flag is applied.

Surrogates

The % recovery of the surrogates is calculated for each sample, blank, and standard. Corrective action is taken whenever one (or more) surrogate recovery from either the base/neutral or acid fraction is outside the acceptance criteria. The following corrective actions are taken when required as stated above:

- Check calculations to assure there are no errors;
- Check internal standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;
- If instrument failure is indicated, reanalyze the sample;

- If more than one method blank surrogate is outside of acceptance criteria or if one surrogate yields less than 10% recovery, then the problem must be corrected before proceeding with sample analysis. This may include reanalysis, reextraction or recalibration;

- If the surrogate could not be measured because the sample required a dilution, no corrective action is required. The recovery of the surrogate is recorded with the note "surrogate diluted out";

- If all QC associated with the sample is within acceptance limits (the method blank surrogate recovery and LCS spike recovery), the problem may be attributed to a matrix effect. Samples exhibiting a matrix effect will be qualified and discussed in the report narrative.

Laboratory Control Sample/Laboratory Control Sample Duplicates

The % recovery of the Laboratory Control Samples and the relative percent difference (RPD) for LCS Duplicates are calculated. Corrective action is taken whenever one (or more) recovery or RPD is outside the acceptance criteria. The following corrective action is taken when required as stated above:

- Check calculations to assure there are no errors;

- Check internal standard and spiking standards [illegible]adation, contamination, etc., and check instrument performance;

- Reanalyze all associated samples, if available;

- If that does not correct the problem, then the data is reported and a qualifying statement regarding the laboratory control sample is included in the report narrative.

For Matrix Spike and Matrix Spike Duplicates, if all QC associated a sample is within acceptance limits (the method blank and LCS/LCS dup spike recovery), the problem may be attributed to a matrix effect Samples exhibiting a matrix effect will be qualified and discussed in the report narrative.

Calibration

For an initial 5 point calibration curve to be valid, the [illegible]elative standard

deviation of the individual relative response factors (RRF) for the Calibration Check Compounds (CCC) should be less than or equal to 40%. If this criteria is not met, then the calibration curve should be reanalyzed.

For continuing calibration checks to be valid, the relative response factor for each of the System Performance Check Compounds (SPCC) should be at least 0.050 and the RRF for each of the CCC should be $\leq$ 40% different from the average RRF from the initial calibration. If these criteria are not met, then the following corrective actions should be taken:

- Check internal standard and standard solutions for degradation, contamination, etc.,
- Check instrument for contamination at the injection port inlet and front end of the column;
- If no source of the problem is identified, then a complete 5 point initial calibration must be performed.

The SPCC and CCC for Semivolatiles are:

SPCC

N-Nitroso-di-n-propylamine
Hexachlorocyclopentadiene
2,4-Dinitrophenol
4-Nitrophenol

CCC

Acenaphthene	4-Chloro-3-Methylphenol
1,4-Dichlorobenzene	2,4-Dichlorophenol
Hexachlorobutadiene	2-Nitrophenol
N-Nitroso-di-phenylamine	Phenol
Di-n-octylphthalate	Pentachlorophenol
Fluoranthene	2,4,6-Trichlorophenol
Benzo(a)pyrene	

15.1.3 Gas Chromatography

Method Blanks

If target compounds are detected in the method blank above the detection limit the corrective action consists of the following:

- checking the calculations;
- reanalyzing the blank;
- flagging the associated sample data;
- Investigating the source of the problem to implement corrective action for the future.

When target compounds are detected in a method blank, the following conditions apply:

- when any target compound is detected in a method blank above the action levels listed earlier, but not in associated samples, then no flag is applied.

Surrogates

The % recovery of the surrogates is calculated for each sample, blank, and standard. Corrective action is taken whenever one (or more) surrogate recovery is outside the acceptance criteria. The following corrective action is taken when required as stated above:

- Check calculations to assure there are no errors;
- Check standard and surrogate solutions for degradation, contamination, etc., and check instrument performance;
- If instrument failure is indicated, reanalyze the sample;
- If the surrogate could not be measured because the sample required a dilution, no corrective action is required. The recovery of the surrogate is recorded with the note "surrogate diluted out";
- If all QC associated with the sample is within acceptance limits (the method blank surrogate recovery and LCS spike recovery), the problem

may be attributed to a matrix effect. Samples exhibiting a matrix effect will be qualified and discussed in the report narrative.

Laboratory Control Sample/Laboratory Control Sample Duplicates

The % recovery of the Laboratory Control Samples and the relative percent difference (RPD) for LCS Duplicates are calculated for each set of spiked samples. Corrective action is taken whenever one (or more) recovery or RPD is outside the acceptance criteria. The following corrective action is taken when required as stated above:

- Check calculations to assure there are no errors;
- Check standard and spiking standard solutions for degradation, contamination, etc., and check instrument performance;
- If that does not correct the problem, then the data is reported and a qualifying statement regarding the laboratory control sample is included in the report narrative.

Calibration

For an initial 5 point calibration curve to be valid, the responses for each compound should be linear over the calibration range. If this criteria is not met, then the calibration curve should be reanalyzed.

For continuing calibration checks to be valid, the % difference in the calibration factor for each compound in calibration should not exceed 15% from the initial calibration. If these criteria are not met, then the following corrective actions should be taken:

- Check standard solutions for degradation, contamination, etc.,
- Check instrument for contamination at the injection port inlet and front end of the column;
- If no source of the problem is identified, then a complete 5 point initial calibration must be performed.

15.1.4 Metals Analyses

Method Blanks

If target analytes are detected in the method blank above the reporting limit the corrective action consists of the following:

- checking the calculations;
- reanalyzing the blank;
- investigating the source if the problem to implement corrective action for the future;
- redigesting and reanalyzing the associated samples if the analyte concentration in the samples is less than 5 times the blank concentration and greater than the reporting limit.
- Data that cannot be regenerated acceptably is flagged as non-compliant.

When target analytes are detected in a method blank, the following data condition applies:

- when any target analyte is detected in a method blank above the action levels listed earlier but not in associated samples, then no flag is applied.

Laboratory Control Sample/Laboratory Control Sample Duplicates

The % recovery of the Laboratory Control Samples and the relative percent difference (RPD) for LCS Duplicates are calculated for each set of spiked samples. Corrective action is taken whenever one (or more) recovery or RPD is outside the acceptance criteria. The following corrective action is taken when required as stated above:

- Check calculations to assure there are no errors;
- Check standard and spiking standard solutions for degradation, contamination, etc., check instrument performance;
- Redigest and reanalyze samples if there is no indication of failure in any of the above;

- If that does not correct the problem, then the data is reported and a qualifying statement regarding the laboratory control sample is included in the report narrative.

An exception to this criteria is allowed for matrix spike samples when the sample concentration exceeds the spike concentration by a factor or 4 or more. In that instance, the data is reported unqualified.

For Matrix Spike, Matrix Spike Duplicates and Sample Duplicates, if all QC associated with a sample is within acceptance limits (the method blank and LCS/LCS dup spike recovery), the problem may be attributed to a matrix effect. Samples exhibiting a matrix effect will be qualified and discussed in the report narrative.

Calibration

For an initial and continuing instrument calibration to be valid, the responses for each analyte must be linear over the calibration range and the accuracy of calibration, as determined by analysis of an independent check standard, must be within $\pm$10% of the true value for ICP/AA analyses and within $\pm$20% for cold vapor AA analyses. If these criteria are not met, then the following corrective actions taken:

- Check standard solutions for degradation, contamination, etc.,
- Check instrument for contamination, incorrect operating conditions, etc.;
- If no source of the problem is identified, then a complete instrument calibration must be performed.

15.1.5 Classical Wet Chemistry Techniques

Method Blanks

If target analytes are detected in the method blank above the detected limit the corrective action consists of the following:

- checking the calculations;
- reanalyzing the blank;

- investigating the source if the problem to implement corrective action for the future;

- redigesting and reanalyzing the associated samples if the analyte concentration in the samples is less than 5 times the blank cor[illegible]ntration.

When target analytes are dete[illegible]d in a method blank, th[illegible]lowing data condition applies:

- when any target analyte is detected in a method bl[illegible]ove the action levels listed earlier but not in associated samples, then no flag is applied.

Labo[illegible]ry Control Sample/Laboratory Control Sample Duplicates

The [illegible]ecovery of the Laboratory Control Samples and th[illegible]elative percent differe[illegible]e (RPD) for LCS Duplicates are calculated for each se[illegible] of spiked samples. Corrective action is taken whenever one (or more) recovery or RPD is outside the acceptance criteria. The following corrective action is taken when required as stated above:

- Check calculations to assure there are no errors;

- Check standard and spiking standard solutions for degradation, contamination, etc., and check instrument performance;

- Reanalyze the sample if no problems are indicated by c[illegible]ng the items above;

- If that does not correct the problem, then the [illegible]s reported and a qualifying statemen[illegible] regarding the laboratory [illegible]mple is included in the report narrative.

An exception to this criteria is allowed for matrix spike samples when the sample concentration exceeds the spike concentration by a factor or 4 or [illegible]. In that instance, the data is reported unqualified.

For Matrix Spike, Matrix Spike Duplicates and Sample Dup[illegible]s, if all QC associated with a sample is within acceptance limits (the [illegible]d blank and

LCS/LCS dup spike recovery), the problem may be attributed to a matrix effect. Samples exhibiting a matrix effect will be qualified and discussed in the report narrative.

Calibration

For an initial and continuing instrument calibration to be valid, the responses for each analyte must be linear over the calibration range and the accuracy of calibration, as determined by analysis of an independent check standard, must be within $\pm$10% of the true value. If these criteria are not met, then the following corrective actions taken:

- Check standard solutions for degradation, contamination, etc.,
- Check instrument for contamination, incorrect operating conditions, etc.;
- If no source of the problem is identified, then a complete instrument calibration must be performed.

15.2 Unusual Occurrences

Whereas out-of-control events involve occurrences outside of pre-established acceptance windows, unusual occurrences are more subjective and involve incidents which may be compliant with the assessment criteria but still warrant investigation. Many of these investigations will be the result of the professional judgement of the analyst, auditor or data reviewer that the analysis was not typical or reasonable. Another example of this type of investigation is an inquiry or questioning of data received from a client or from the results of performance evaluation samples.

FIGURE 15-1

PACE NE CORRECTIVE ACTION REPORT

Revision 1 - 12/92

Protocol_____

Date:_____ Reported By:_____

Sample ID Number(s) Involved and QC batch #:_____

Type of Event: ☐ Out-of Control Event ☐ Unusual Occurrence

Description of Event:_____

Discussion of Known or Suspected Cause:_____

Corrective Action(s) Taken (include date, person and action):_____

Signed_____

Signed_____
Group Supervisor

Signed_____
Technical Director

Follow Up:_____

Quality Assurance Review

_____ Date _____

Management Review

_____ Date _____

16.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The Quality Assurance Officer and Quality Assurance Auditors are responsible for preparing reports to management indicating effectiveness of the laboratory Quality Assurance Program.

16.1 Quality Assurance Auditors

Results of audits performed by the QA staff are detailed in formal, written audit reports. These reports are distributed to the audited personnel, section supervisor, Laboratory director, QA officer, and Regional Director for review and appropriate action. These and other QA-related reports are distributed as produced, with no set schedule.

Auditor reports will include, but not be limited to:

- Results of internal laboratory review activities
- Results of internal data review activities
- Results of Proficiency Evaluation studies
- Results of state certification applications
- Summary of holding time exceedence and data qualification
- Method detection limit study status

To demonstrate management review, the audit report will contain a page which will be signed and dated by the QA Officer and Regional Director acknowledging that they have received the report and have reviewed its contents, and taken the necessary action dictated by their position.

16.2 Quality Assurance Officer

The Quality Assurance Officer will issue a report of QA activities and findings on a monthly basis to the Regional Director. The monthly status report will include:

- Results of internal systems or performance audits
- Corrective Action recommendations
- Discussion of QA issues raised by laboratory users
- Results of third party or external audits
- Status of laboratory certifications
- Other significant events
- Performance Evaluation Sample Results

16.3 Management Review of the Quality Assurance Program

Review of the appropriateness and adequacy of the Quality Assurance Program is ongoing. At anytime, any laboratory employee, through the Laboratory Manager may present recommended changes to the Quality Assurance Officer.

During system audits, the Quality Assurance Program should be discussed. The audit report will document recommendations made by either the Laboratory Manager or the auditor for revision.

#161

EXCELLENCE
IN ANALYSIS

(Pace)

COAST-TO-COAST ANALYTICAL SERVICES, INC.
INDIANAPOLIS DIVISION
QUALITY ASSURANCE
PROGRAM PLAN
FOR
ENVIRONMENTAL CHEMICAL MONITORING

Prepared by:

(Pace)

Coast-To-Coast Analytical Services, Inc.
Indianapolis Division
7726 Moller Road
Indianapolis, Indiana 46268

Initial Revision 1.0 dated July, 1992

Revision 3.0
January, 1994

Approval:

Julia K. Tillman 1-31-94

Julia K. Tillman, QA/QC Officer

01-31-94

Stephen A. Barnett, President

CONFIDENTIAL

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1. INTRODUCTION

Based in Indianapolis, CCAS (hereafter referred to as CCASI) is a full service laboratory, specializing in dioxin/furan analysis, that provides quality environmental analyses. These services are provided to many industrial clients as well as governmental agencies, public utilities, engineering firms, and waste management companies. It is one of only a few firms across the country to have successfully participated in the Environmental Protection Agency's Contract Laboratory Program for the testing of environmental matrices for dioxins/furans. CCASI has achieved national recognition in the environmental industry by offering high levels of professional service, meeting or exceeding stringent quality standards, and using the most advanced instrumentation.

The U.S. EPA requires that each laboratory generating data implement minimum standard operating procedures which assure that the precision, accuracy, completeness, and representativeness of its data are known and documented. This document describes the CCASI Quality Assurance policies and procedures related to chemical monitoring for environmental pollutants.

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2. QUALITY ASSURANCE POLICY

In order to achieve our corporate objective, CCASI has established an extensive Quality Assurance (QA) program which combines good laboratory practices, method compliance, and quantitative criteria for data acceptability. Compliance with the QA plan presented herein provides the foundation for technically sound analytical results of known and documentable quality. This program relies on clearly defined objectives, well documented procedures, a comprehensive audit system, and management support for its effectiveness. Every employee at CCASI has a role in quality control, and every employee is responsible and accountable for the quality of their work. Therefore, it is our requirement that all laboratory personnel read, review, and understand the procedures and requirements established by this document.

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3. PURPOSE AND SCOPE

PURPOSE

This QA Program Plan presents an overview of the essential elements of the CCASI QA program. CCASI has modeled this plan along EPA guidelines as outlined in "Guidelines and Specifications of Preparing Quality Assurance Program Plans (QAPPs) and Quality Assurance Annual Report and Workplans (QAARWs) for EPA National Program Offices (NPOs) and the Office of Research and Development (ORD)", September 1987, and "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans", QAMS-005/80, December 29, 1980. Both of these documents have been issued by the Office of Monitoring Systems and Quality Assurance, Office of Research and Development, U.S. EPA.

The purpose of this document is to establish standard operating procedures to ensure that all data generated in the laboratory conform to specific requirements for accuracy, precision, and completeness, as related to the appropriate type of analysis, analyte, and matrix. This quality assurance/quality control plan describes the organization and procedures routinely incorporated into all analyses performed

by CCASI for the purpose of producing quality, reliable data.

SCOPE

The CCASI QA program is designed to control and monitor the quality of data generated in the laboratory. The program has four key elements. These elements entail:

- Providing information to demonstrate the overall qualifications and capability of the laboratory to perform environmental analyses;
- Establishing procedures to measure the laboratory's performance on a daily basis;
- Measuring matrix effects to determine the effect of a specific matrix on method performance; and
- Reporting appropriate QC information with the analytical results to enable the end-user to assess the quality of the data.

The specific procedures involved in implementing each aspect of the CCASI QA program are described in this document. The QA/QC policies and procedures described in this document are designed to eliminate systematic errors and minimize the occurrence of other errors. However, no QA program, regardless of how elaborate, can eliminate all errors which may occur during an analysis. The QA program builds the framework for eliminating, to the extent possible, systematic and random errors, and identifying and correcting those

errors which do occasionally occur. These QA/QC policies and procedures must be coupled with the sound, professional judgment of the technical staff in interpreting the events surrounding the generation of final results in order to ensure that analytical data produced is of acceptable, known, and documentable quality.

The management staff at CCASI is committed to ensuring that the quality, integrity, technical validity, and defensibility of analytical results are of paramount importance to every member of this organization.

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4. DEFINITION OF TERMS

Quality Assurance (QA): the total integrated program for assuring the reliability of data generated in the laboratory.

Quality Control (QC): the routine application of specific, well documented procedures to ensure the generation of data of known and acceptable quality, thus fulfilling the objectives of the QA program.

Quality Assurance Program Plan (QAPP): an assemblage of management policies, objectives, principles, and general procedures outlining the techniques by which the laboratory produces data of known and acceptable quality.

Standard Operating Procedure (SOP): a detailed, written description of a procedure designed to systematize and standardize the performance of the procedure.

Quality Assurance Project Plan (QAPjP): an assemblage of detailed procedures describing how the laboratory will generate data that meet the Data Quality Objectives (DQOs) of a specific project.

Holding Time: the period of time during which a sample can be stored after collection and preservation without

significantly affecting the accuracy of the analysis.

Analytical Holding Time: the period of time during which a sample extract can be stored before analysis is completed without significantly affecting the accuracy of the analysis.

Sample Delivery Acceptance: the point in time at which CCASI determines that it can proceed with the analytical work. Sample Delivery Acceptance follows receipt and inspection of the samples and complete definition of analysis required.

Initiation of Preparation: the point in time at which the separation of organic or inorganic extractable analytes from the sample matrix by appropriate extraction media is initiated.

Initiation of Analysis: the point in time at which the extracted analytes are introduced into an instrument or to a procedure which complies with the SOP for analysis of the parameter of interest.

5. RESPONSIBILITIES AND AUTHORITIES

We believe that the success of CCASI is dependent upon the continued commitment of all within the organization to a strong and viable QA Program. The following section outlines responsibilities, levels of authority, and also includes an organizational chart. Resumes are available upon request.

The QA effort within CCASI is directed by the QA/QC Officer who reports directly to the President of CCASI. The responsibilities of the QA/QC Officer are as follows:

- Developing and implementing a QA program that ensures that all data generated by CCASI is scientifically sound, legally defensible, and of known precision and accuracy;
- Monitoring the QA Plan to ensure compliance with QA objectives in the laboratory;
- Developing and implementing new QA procedures within the corporation to improve data quality;
- Conducting audits and inspections of the laboratory on a regular basis, reporting the results of those audits to management, and applying corrective actions as needed to ensure compliance with the CCASI QA Plan;
- Serving as the in-house client representative on all project inquiries involving data quality issues;
- Assisting laboratory personnel in the writing, reviewing, and implementation of SOPs;
- Assuring that the laboratory staff has access to current SOPs;

- Reviewing all data generated by CCASI to ensure compliance with protocols being used; and
- Maintaining all documents, files, and records pertaining to QA.

The QA/QC Officer is the final authority within each department on all issues dealing with data quality. This also includes having the final authority to accept or reject data that does not meet QA/QC criteria as outlined in methods or SOPs. He/She has the authority to make recommendations to management regarding procedures that may need to be amended or discontinued, or analyses that should be suspended or repeated.

The President of CCASI is responsible for the following:

- Monitoring the implementation of the QA Plan within the laboratory to ensure complete compliance with QA objectives;
- Prescribing, when necessary, and monitoring corrective actions; and
- Promoting sound QA practices within the environmental regulatory and analytical communities.

The President is involved in making the final decisions dealing with quality, and he/she has the authority to require that procedures be amended or discontinued, or analyses suspended or repeated. Also, the President has the authority to suspend or terminate employees on the grounds of dishonesty, incompetence, or repeated non-compliance with QA

procedures.

The Operations Manager is responsible for coordination of all technical and analytical activity in the laboratory. He/She is directly accountable to the President of CCASI. Other QA responsibilities include:

- Actively supporting the implementation of the CCASI QA Plan within the laboratory;
- Maintaining accurate SOPs and ensuring their use in the laboratory;
- Maintaining a work environment that emphasizes the importance of data quality; and
- Prescribing and monitoring corrective actions.

The Operations Manager has the authority to accept or reject data based on compliance with well defined QC criteria. In addition, the Operations Manager can accept or reject data that falls outside of established QC guidelines if, in his/her judgment, there are technical reasons which warrant the acceptance or rejection of the data. However, in this case, the QA/QC Officer must make the final ruling. These circumstances must be well documented, and any need for corrective action identified by the incident must be defined and initiated.

The Operations Manager is also involved in making the final decision on all issues dealing with quality, and he/she has

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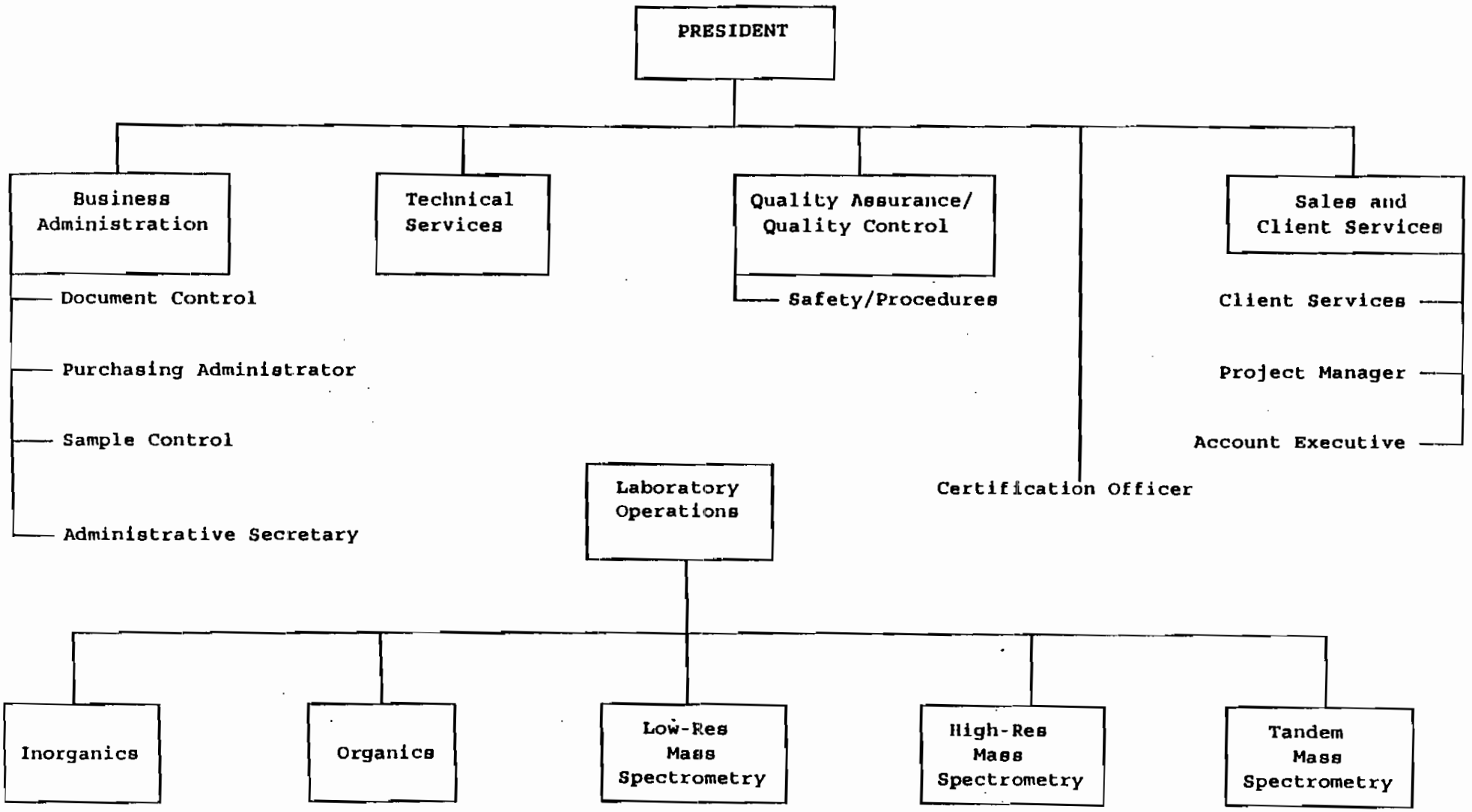
the authority to require that procedures be amended or discontinued, or analyses suspended or repeated. The Operations Manager has the authority to suspend or terminate laboratory employees only on the grounds of dishonesty, incompetence, or repeated non-compliance with QA procedures.

All other laboratory personnel involved in the generation and reporting of data have a responsibility to understand and follow the CCASI QA Plan. Other QA responsibilities include:

- Having a working knowledge of the CCASI QA Plan;
- Ensuring that all work is generated in compliance with the CCASI QA Plan;
- Performing all work according to written SOPs or methods;
- Ensuring that all documentation related to the work is complete and accurate; and
- Providing management with immediate notification of quality problems.

Laboratory personnel have the authority to accept or reject data based on compliance with well defined QC criteria. The acceptance or rejection of data that falls outside of established QC guidelines must be approved by laboratory management and the QA/QC Officer. The authority of the laboratory personnel flows from the Operations Manager.

COAST-TO-COAST ANALYTICAL SERVICES, INC.
INDIANAPOLIS DIVISION



6. FACILITY DESCRIPTION

CCASI occupies a 13,000 square foot facility located in Indianapolis, Indiana, approximately 20 minutes north of the airport. The facility contains separate/dedicated laboratory preparation rooms with sophisticated air handling and exhaust systems, three dedicated mass spectrometry laboratories, an inorganic laboratory, dedicated glassware cleaning area, and document control areas.

CCASI has the capability to perform standard GC/MS, GC/HRMS, GC, HPLC, ICP, and AA methods as well as a variety of specialized tandem MS/MS methods. The laboratory staff includes low resolution, high resolution, and tandem mass spectrometer operators, ICP and AA operators, and staff members who are experienced in organic and inorganic chemistry procedures and sample preparation.

Laboratory equipment available includes gas chromatographs, liquid chromatographs, spectrophotometers, ICP, AA, and support equipment. The principle laboratory instrumentation includes a tandem quadrupole mass spectrometer, two E-B-E geometry computer-controlled double-focusing mass spectrometers, and several conventional Hewlett-Packard GC/MS

systems.

The tandem quadrupole mass spectrometer is a SCIEX TAGA 6000E. The instrument is equipped with extended mass range to 1500 amu, and all available interfaces including GC, APCI, LPCI, LC, probes, and gas sampling. The instrument is computer-controlled, and it is capable of all forms of MS/MS operation, including SIM, parent/daughter SIM, parent scans, daughter scans, and neutral loss scans. The instrument is cryopumped and capable of handling continuous gas loads directly into the source as high as 40 ml/min. The low pressure CI source is a bright Townsend discharge that generates an extremely high ion density.

The two high resolution mass spectrometers are VG AutoSpec hybrid magnetic sector instruments employing E-B-E geometry. The data systems used are DEC VAXstation 3100 workstations running OPUS data analysis and chemical information software under the VMS operating system. The present configuration is for GC or probe sample introduction into an EI source. The system is capable of reconfiguration to accept other means of sample introduction (i.e. HPLC) and other ionization modes (i.e. CI, thermospray, FAB, etc.).

The conventional Hewlett-Packard GC/MS systems are capillary

gas chromatographs coupled with mass selective detectors (MSDs). The data system supporting the three HP5890/HP5970 GC/MSD units is an HP 1000 A-Series computer, Real-Time Executive (RTE-A) operating system with HP mass spectrometer application/data reduction software and HP peripheral hardware (scanning interface, disk drives, terminals, printers, etc.). It controls or assists in instrument tuning, data acquisition, data reduction, report production, and automation.

In addition to the HP5970/RTE systems, an HP5890/HP5971A GC/MSD is linked to an MS-DOS ChemStation running custom, proprietary software developed by Hewlett-Packard and CCASI. This system provides additional flexibility in automated report generation.

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7. SAMPLING PROCEDURES

The generation of quality data begins with the collection of the sample, therefore the integrity of the sample collection process is of concern to the laboratory. In order to help ensure sample integrity, a few points must be considered:

- Samples must be collected in appropriate containers - in general, pre-cleaned glass containers are used for organic parameters, and pre-cleaned polyethylene containers are used for inorganic/metal parameters;
- The sample containers must be properly cleaned to ensure that the sample is not contaminated during the collection process;
- Samples must be preserved appropriately to minimize the loss of compounds of interest due to adsorption, chemical or biological degradation, or volatilization;
- Appropriate volumes of sample must be collected to ensure that the required detection limit can be met and quality control samples can be analyzed;
- Samples must be properly shipped to the laboratory in the appropriate time frame to ensure that holding times for the analyses can be met.

Although CCASI does not perform sampling, we can assist in the sample collection process by providing coolers, appropriate sample containers that are properly cleaned, and proper preservatives for use in sample collection. Appropriate containers and preservatives, and minimum sample volumes required for analyzing routine organic, metal, and

conventional parameters are listed in Tables 1 - 8. These tables may be found in Appendix I of this document. Sample collection guidelines are listed in Appendix II. The collection guidelines are supplied with every sample kit that is requested. It is important that samples are collected properly and preserved properly at the time of collection, for the quality of the data produced can be no better than the quality of the sample supplied to the laboratory.

Holding Times

EPA has established holding time requirements for some analyses. These holding time requirements are listed in Tables 1 - 8 of Appendix I. As indicated, holding time requirements differ depending on the regulatory program. The majority of the work done by CCASI is in support of RCRA and CERCLA/Superfund activities. Therefore, CCASI follows the holding times given in SW-846 (3rd. Edition) unless otherwise instructed by the client. CLP holding times are followed when CLP protocols are requested by the client. Other holding times will be honored if arrangements are made with CCASI prior to sample receipt.

CCASI is obligated to initiate preparation and/or analysis of samples within holding times if sample delivery acceptance

occurs within 72 hours of sampling, or before one-half of the holding time period has expired, whichever is less. In the event that samples are received with more than half the holding time already expired, CCASI will make every effort to analyze samples before holding times have completely expired. If necessary, clients may arrange for rush service, prior to sample receipt by the laboratory, in order to ensure that holding times are not exceeded. Occasionally, a sample must be re-analyzed to comply with the requirements documented in this QA Program Plan. If this re-analysis is conducted outside of holding time, CCASI will be considered to have fulfilled its obligation to meet holding times if the first preparation and/or analysis was initiated within the prescribed holding time.

Please note that the volume requirements, preservation protocols, container types, and holding times listed in Tables 1 - 8 of Appendix I may vary depending upon the requirements of given programs, matrices, special methods, clients, or governmental agency protocols. CCASI personnel can provide consultation and assistance in this regard should such assistance be required.

8. SAMPLE CUSTODY

An organized and efficient sample management system is a necessary and critical foundation on which actual analyses of samples are based. Sample management includes sample receipt, chain-of-custody documentation, sample preservation documentation, sample storage, case file creation, reporting and invoicing, and sample retention and disposal.

Sample Receipt

Samples received at CCASI are inspected for integrity, and field documentation is reviewed for accuracy and completeness. If chain-of-custody forms do not accompany the samples, the sample custodian will initiate these forms. When samples are received with missing or deficient chain-of-custody forms, the legal traceability of these samples can not extend to the time of collection, but must begin at the time of laboratory receipt.

Chain-of-custody and sample integrity problems are noted and recorded during sample log-in. Client Services is informed of the deficiencies and will advise the laboratory on the desired disposition of the samples after contacting the client. The sample is then assigned a unique sample number

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which will identify the sample in the Laboratory Information Management System (LIMS). Reference to a sample in any communication will include the laboratory assigned sample ID number and/or the client sample ID number to specify which sample is of concern.

Chain-of-Custody

(C.O.C.) procedures document the history of samples and constitute a crucial part of sampling and analysis programs. C.O.C. documentation assists and enables the identification and tracking of a sample from the time of collection through the time of analysis.

All samples which are received at CCASI must be accompanied by a C.O.C. The C.O.C. should include the following data:

- Date of Sampling
- Sample Identification
- Sample Description
- Client/Project Identification
- Number of Containers Per Sample
- Preservation (if present)
- Analysis Required
- Special Instructions/Notes
- Signature of Sampler
- Time/Date of Transfer
- Signature of Recipient

Sample containers should include the following data:

- Date of Sampling
- Sample Identification/Description
- Analysis Required

- Client/Project Identification

When samples are received at CCASI, the Sample Custodian will verify each and every sample against the C.O.C. forms, note any discrepancies or losses of samples, and then sign for receipt of the samples. The pH and temperature of each water sample received by CCASI will be taken at the time samples are logged into the LIMS, and this information will be recorded on the C.O.C. and into the LIMS. If samples are hand delivered by the client, a copy of the signed C.O.C. will be given to the client at that time. In the event that samples are shipped to the laboratory with incomplete or ambiguous documentation, a call to the client will be made by Client Services to confirm analysis. A signed copy of the C.O.C. and a copy of the LIMS Confirmation will be sent to all clients within 72 hours of receipt. In the event that sample results are needed on a rush basis or samples are submitted for rapid-turnaround dioxin analysis, the C.O.C. and LIMS Work Order Confirmation copies will not be sent to the client. Samples will remain under the control of the Sample Custodian until samples are transferred to the appropriate storage area or to the laboratory staff for processing. Following transfer to the laboratory staff, all samples must remain in custody, and such custody must be

documented.

A sample is considered to be in custody if it:

- Is in the physical possession of the responsible party; or
- Is in view of the responsible party; or
- Is secured by the responsible party to prevent tampering; or
- Is secured by the responsible party in a restricted area.

Sample Preservation/Bottle Preparation

In most cases, sample bottles and sample preservation will be the responsibility of the client. In addition, sample homogenization will also be the responsibility of the client. In cases where information is sought regarding bottling, preservation, and/or shipping, CCASI will advise according to the most recent recommendations from the U.S. EPA or other relevant federal/state regulatory authorities as applicable, or CCASI will provide references for other consultation or assistance.

Sample bottles or other appropriate containers can be provided to the client by CCASI if reasonable notice is given prior to the time needed. It is the policy of CCASI that these containers will be supplied at no additional cost provided that samples are submitted to CCASI for analysis

within thirty (30) days of supply date. In the event that samples are not received by CCASI within this period, and alternate arrangements have not been made, the cost of the containers and any additional items sent (i.e. ice packs, coolers, etc.) will be invoiced to the client. Also, if the number of samples received is less than 75% of the number of containers supplied, the client will be invoiced for the difference in number of remaining, unused containers.

Sample Storage

Samples will be stored in a secured area of the type dictated by the analytical methodology. For refrigerated areas, the Sample Custodian will monitor and record the temperatures twice daily. Sample fractions and extracts will also be stored under the conditions dictated by the analytical methodology.

Case File

Upon receipt of samples, CCASI will create a case file in which to maintain records associated with the project. In addition to administrative information, the requested analyses and any requested modifications will be included in the file. As the project progresses, C.O.C. forms, tracking

sheets, change orders, telephone conversation logs, analytical results, and any other pertinent information will be added to the file.

Reporting and Invoicing

CCASI offers three (3) levels of report formats depending on how much detail is needed by the client. Level I reports are basically "results only" reports; Level II reports include continuing calibration summaries and sample chromatograms; and Level III reports are "CLP type" reports which include all paperwork and all data generated. Invoices are included with a copy of the final report mailed to clients, or they are sent simultaneously if the billing address differs from the reporting address.

Sample Retention and Disposal

In the case of contracts between CCASI and the U.S. EPA or other party, samples and sample extracts will be retained for the specified time as outlined in the original contract. At the end of this time, packing materials and shipping containers will be returned, and samples and sample extracts will be disposed of in an appropriate manner.

In all other cases of non-dioxin/furan analyses conducted for

private clients, the packing materials and shipping containers will be returned to the client upon verification of the data and transmission of the final data package. Samples will be disposed of in an appropriate manner by CCASI. Unused portions of solid matrix samples received for dioxin/furan analysis will be returned to the client for disposal. Aqueous samples received for dioxin/furan analysis are used up entirely in the extraction process, so CCASI will appropriately dispose of those sample containers.

9. CALIBRATION PROCEDURES AND FREQUENCY

Standard/Reagent Preparation

A critical element in the generation of quality data is the purity/quality and traceability of the standard solutions and reagents used in analytical operation. Whenever possible, primary reference materials will be obtained from the National Institution of Standards and Technology (NIST). In the absence of available reference materials from the NIST, other reliable sources will be sought. These reference materials will be used for instrument calibration, quality control spikes, internal standards, and/or performance evaluations. Secondary reference materials may be used for these functions provided that they are traceable to a NIST standard, or they have been compared to a NIST standard within the laboratory.

Stock and working standards are checked regularly for signs of deterioration, such as discoloration, formation of precipitates, or change in concentration. Care is exercised in the proper storage and handling of standard solutions, and all containers are labeled as to compound, concentration, solvent, expiration date, and preparation data. All

standards or standard solutions are validated prior to use. This validation may take the form of supplier certifications. This validation may also be restandardization for acids or bases, response factor comparison, standard curve response, or comparison to other standards made at a different time or by a different analyst.

Laboratory reagents will be of the highest quality obtainable to minimize or eliminate background concentration of analytes to be measured. Also, reagents must not contain other contaminants that will interfere with the analytes of interest. Each new lot of solvent (high volume solvents only) is analyzed prior to use to ensure the absence of interfering constituents. Reagents are also monitored through the preparation and analysis of method blanks with each batch of samples and on a quarterly basis for purity.

Instrument Calibration and Tuning

Before any instrument can be used as a measurement device, the instrumental response to known reference materials must be determined. The manner in which the various instruments are calibrated will be dependent on the particular instrument, and the intended use of the instrument. These calibrations will be based upon the strictest adherence to

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the guidelines published by the U.S. EPA or other relevant Federal/State regulatory authority for the intended analysis. All sample measurements will be made within the calibrated range of the instrument. Measurements outside this range will be flagged as such, or the extract will be diluted and re-analyzed.

Gas Chromatography/Mass Spectrometry (GC/MS) - Volatile/Semivolatile Organics

Each day, prior to analysis of samples or standards, the instrument is tuned with bromofluorobenzene (BFB) for volatile compounds and decafluorotriphenylphosphine (DFTPP) for semi-volatile compounds (according to the tuning criteria specified in the U.S. EPA method being utilized). No samples will be analyzed until the instrument has met tuning criteria. Once these criteria are met, the mass spectrometers are considered to be in compliance for the time period stated in the method after the injection of the tuning compound. Any analytical run which lasts longer than the stated period must include a tune check at or before expiration of the previous tune check.

The instrument is then calibrated for all target compounds. The GC/MS systems must be initially calibrated at a minimum

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of 5 concentrations to determine the linearity of response. Volatile standards are run at 20, 50, 100, 150 and 200 ug/L. Semi-volatile standards are run at 20, 50, 80, 120, and 160 ug/L. Once these calibration curves are produced, certain key compounds referred to as System Performance Calibration Compounds (SPCC) and Continuing Calibration Compounds (CCC) are evaluated every 12 hours to ensure that the system remains calibrated. If the standard does not meet the established criteria, the system is recalibrated.

Gas Chromatography/Tandem Mass Spectrometry (GC/MS/MS) - TCDD/TCDF

The initial calibration consists of triplicate injections of three solutions with varying concentrations of native analyte. Acceptance of the initial calibration is based upon the RSD of all native RRFs (Relative Response Factors) which must not exceed ten percent. The mean ion ratio is also determined by the initial calibration. Upon acceptance of the initial calibration, the instrument is available for use providing a daily calibration check verifies the initial calibration. The daily calibration check is a single injection of the low concentration calibration solution (Cal I). The ion ratio must fall within plus or minus ten percent of the mean, and the RRFn must fall within plus or minus ten

percent of the mean as set by the initial calibration. If all criteria are satisfied, the instrument is considered to be in compliance for a period of 12 hours. Analytical runs which last longer than 12 hours must include another daily calibration check at or before 12 hours from the previous daily calibration check. The initial calibration is valid for a period of up to one year providing that the daily calibration checks will verify the initial calibration for that long.

A blank calibration is performed to determine a correction factor for the internal standard contribution to the native analyte responses. The correction factor is used to subtract this response from every sample result.

Gas Chromatography/Mass Spectrometry (GC/MS) - Low Resolution PCDD/PCDF

Once per week (or as required), the instrument is tuned with perfluorotributylamine (PFTBA or FC43) in order to establish proper mass assignment and resolution. Upon completion, the instrument must be calibrated via initial calibration procedures, or the initial calibration must be validated via the daily calibration check.

The initial calibration consists of single injections of five

solutions with varying concentrations of native analytes. Acceptance of the initial calibration is based upon the following three factors:

(1) The signal-to-noise ratio for all analytes must be greater than or equal to 10%;

(2) Ion ratios for all analytes must fall within the specified ranges as documented in the applicable EPA Method or Statement of Work (SOW); and

(3) Relative Standard Deviations (RSDs) for all Relative Response Factors (RRFs) must be less than or equal to 15%.

Upon acceptance of the initial calibration, the instrument is available for use providing a daily calibration check verifies the initial calibration. The daily calibration check is composed of a single injection of the middle calibration solution (CC3). The signal-to-noise ratio, ion ratio, isomer specificity requirements, and RRF deviation must meet the criteria specified in the applicable and current EPA Method or SOW. If all criteria are satisfied, the instrument is considered to be in compliance for a period of 12 hours. Analytical runs which last longer than 12 hours must include another daily calibration check at or before 12 hours from the previous daily calibration check.

An ending calibration is required as the final analysis, and it should be analyzed within 12 hours from the last daily

calibration check. This final calibration is composed of a single injection of either CC1 or CC3, depending on the methodology requirement. Criteria specified for this analysis must be met prior to acceptance of data from the analytical run.

High Resolution Gas Chromatography/Mass Spectrometry (HRGC/HRMS) - PCDD/PCDF

On a daily basis, mass calibration and resolution must be established prior to initial and/or daily calibration. These criteria are established with the reference compound perfluorokerosene (PFK). Both mass calibration and resolution checks are valid for a period of 12 hours. If analytical runs last longer than 12 hours, the mass calibration and resolution must be checked again at or before 12 hours from the previous mass calibration and resolution measurements.

Initial calibration is established using single injections of five solutions with varying concentrations of native analyte. Ion abundances, signal-to-noise ratios, and isomer specificity must meet the criteria established by the current method or SOW. Additionally, the relative standard deviation of each analyte's RRF must meet the criteria specified within

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the current EPA method or SOW prior to acceptance. At the beginning of each 12 hour shift, system and calibration performance is verified via the analysis of Continuing Calibration 3 (CC3) solution. Ion abundance ratios, signal-to-noise ratios, isomer specificity, and percent deviation of the RRFs must fall within the criteria specified in the current EPA method or SOW. Additionally, the retention times (absolute and relative) of the various analytes must meet the defined criteria as specified in the EPA method or SOW.

Resolution must be demonstrated at the beginning and end of the 12 hour analytical window. The initial resolving power must meet the specifications outlined in the current EPA method or SOW.

Chromatography

The field of chromatography involves a variety of instrumentation and detection systems. While calibration standards and acceptance criteria vary depending on the type of systems and analytical methodology required for a specific analysis, the general principles of calibration apply uniformly. Each chromatographic system is calibrated prior to performance of analysis. Initial calibration consists of

determining the instrument's linear range, establishing limits of detection, and establishing retention time windows. The calibration is checked on a daily basis to ensure that the system remains within specifications. If the daily calibration check does not meet established criteria, the system is recalibrated, and samples analyzed since the last acceptable calibration check are re-analyzed.

Metals

All procedures utilizing atomic spectroscopy (Graphite Furnace Atomic Absorption {GFAA}, or Cold Vapor Atomic Absorption {CVAA}) or Inductively Coupled Argon Plasma (ICAP) must be calibrated daily to determine and verify linear calibration ranges as well as analytical sensitivity and detection limits.

The ICP is calibrated prior to any analysis according to criteria prescribed in the protocol being utilized. The calibration is then verified using standards from an independent source. The linear range of the instrument is established once every calendar quarter using a linear range verification check standard. No values are reported above this upper concentration value without dilution.

A calibration curve is established daily by analyzing a minimum of two standards, one of which is a calibration blank. The calibration is monitored throughout the day by analyzing a Continuing Calibration Verification standard (CCV). The standard check must meet established criteria, or the system is recalibrated, and all samples analyzed since the last acceptable calibration check are re-analyzed.

An interelement check standard is analyzed at the beginning and end of each analytical run on a continuing basis to verify that interelement and background correction factors have remained constant. Results outside of the established criteria trigger re-analysis of samples.

The AA is calibrated prior to any analysis being conducted. A calibration curve is prepared with a minimum of a calibration blank and three standards, and it is then verified with a standard that has been prepared from an independent source at a concentration near the middle of the calibration range. The calibration is then validated periodically during analysis with a midpoint calibration standard. If the ongoing calibration standard does not meet established acceptance criteria, the system is recalibrated, and all samples analyzed since the last acceptable

calibration check are re-analyzed. All samples are spiked to verify the absence of matrix effects or interferences. The method of standard additions is used when matrix interferences are present.

General and Physical Chemistry

The calibration procedures for general chemistries (i.e. titrations, colorimetric tests, gravimetric procedures, etc.) are included in the methods used. In general, all procedures must be verified and/or equipment calibrated prior to use. Calibration consists of defining the linear range by use of a series of standard solutions, establishing limits of detection, and identifying potential interferences. The calibration is checked periodically during the analysis to confirm that the system remains within specifications. If the check should fall outside of acceptance limits, the system will be recalibrated, and all samples analyzed since the last acceptable calibration check will be re-analyzed.

10. ANALYTICAL PROCEDURES

Analytical methods are routinely conducted as outlined in published sources. Modifications to these methods may be necessary in order to provide accurate analysis of particularly complex matrices. When modifications to standard analytical methods are required, the specific alterations, as well as the reason for the change, will be attached to the printed method. Generally, the methods used are those specified by the U.S. EPA and other federal agencies, state agencies, and professional organizations as provided in the following references:

- "Methods for Chemical Analysis of Water and Wastes", EPA-600/4-79-020 (revised March, 1983).
- Current EPA (CLP) protocols for the analysis of organic and inorganic hazardous substances including dioxins and furans..
- "Test Methods for Evaluating Solid Waste", (SW-846), 3rd Edition (1986), Update I (1989), Final Update I (1992), Office of Solid Waste and Emergency Response, U.S. EPA.
- "Standard Methods for the Examination of Water and Wastewater", 17th Edition, American Public Health Association, American Water Works Association, Water Pollution Control Federation, Washington, D.C. (1989).
- "Annual Book of ASTM Standards", Volumes 11.01, 11.02 11.03, 11.04, and 14.02, American Society for Testing and Materials (ASTM), Philadelphia, PA (1990).

- "Guidelines Establishing Test Procedures for the Analysis of Pollutants", 40 CFR, Part 136 (Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977), July 1, 1991.

The choice of methodology is dependent upon the objectives of the study in terms of qualitative certainty, quantitative sensitivity, precision and accuracy, and the type of matrix to be analyzed. Each method used routinely is documented in the form of an SOP. The SOP contains detailed instructions concerning both the use and the expected performance of the method. Any deviations from published methodology are documented and explained in the SOP. A description of the contents of laboratory SOPs is given in section 18 of this document.

Before any methods are routinely used to generate analytical data, the method is validated. Validation criteria consists of:

- Method selection by a senior staff member;
- Documentation of the method in an SOP - this includes a summary of the method, detailed description of the analytical procedure, calculations, reporting formats, safety concerns, and special remarks;
- Testing of the method to verify detection limits and linear range, establish reporting limits, determine precision and accuracy criteria; and
- Establishment of data acceptance criteria that must be approved by a senior staff member and the QA/QC Officer.

11. DATA REDUCTION, VALIDATION, AND REPORTING

Data Reduction and Validation

All analytical data generated by CCASI are extensively checked for accuracy and completeness. The data validation process consists of data generation, reduction, and review.

The analyst who generates the analytical data has the prime responsibility for the correctness and completeness of the data. All data is generated and reduced following protocols specified in laboratory SOPs. Each analyst reviews the quality of his or her work based on an established set of guidelines. The analyst reviews the data package to ensure that:

- Sample preparation information is correct and complete;
- Analysis information is correct and complete;
- The appropriate SOPs have been followed;
- QC samples are within established control limits;
- Blanks are within appropriate QC limits;
- Special sample preparation and analytical requirements have been met; and
- Documentation is complete (i.e. all anomalies in the preparation and analysis have been documented, out-of-control situations are resolved, holding times are documented, etc.).

Following this initial review, the analyst then passes the data package to an independent reviewer, who performs the first level of independent review.

Level 1 review is performed by the Group Leader. This is designed to be an independent review of the data and is structured to ensure that:

- Calibration data are scientifically sound, appropriate to the method, and completely documented;
- QC samples are within established guidelines;
- Qualitative identification of sample components is correct;
- Quantitative results are correct;
- Documentation is complete and correct (i.e. all anomalies in the preparation and analysis have been documented, out-of-control situations are resolved, holding times are documented, etc.);
- The data is ready for incorporation into the final report; and
- The data package is complete and ready to be archived.

An important element of Level 1 review is the documentation of any errors that have been identified and corrected during the review process. Errors that are found are documented and transmitted to the appropriate person. The cause of the error is then addressed with additional training or clarification of procedures to ensure that quality data will be generated at the bench. Once any errors have been

corrected, the data package is then approved for release and a final report is prepared.

The Level 2 review is performed by the Operations Manager to ensure that the data meets the overall needs of the client. The primary purpose of this review is, at a minimum, to track holding times, frequency of reruns, and turnaround times. The Operations Manager will document trends in these areas and recommend corrective action if necessary.

After approval by the Operations Manager, the package then goes to the QA/QC Officer. This Level 3 review is to ensure compliance with QA/QC criteria as established by the method. The QA/QC Officer will check items such as calibrations, surrogates, internal standards, matrix spikes or lab control samples, and blanks. If errors or deficiencies are found, they will be documented, and the package will be returned to the Operations Manager who will return it to the appropriate person for correction.

The fourth level of review is conducted by the Document Control Officer. At this point, the package will again be checked for completeness, transcription errors, and overall appearance.

Each step of this review process involves evaluation of data quality based on both the results of the QC data and the professional judgment of those conducting the review. This application of technical knowledge and experience to the evaluation of the data is essential in ensuring that data of high quality is generated consistently. Each level of review is clearly documented within the package.

Data Reporting

A variety of reporting formats, from computerized data tables, to complex reports discussing regulatory issues, to CLP deliverables packages are available. In general, CCASI reports include:

Case Narrative: Description of sample types, test(s) performed, dates received, any problems encountered, and general comments are given.

Analytical Data: Data is reported on summary sheets by test. Pertinent information including dates received, prepared or extracted, and analyzed are listed. The reporting limit for each non-dioxin/furan analyte is also given.

QC Information: The results (Percent Recovery) of the Laboratory Control Samples analyzed with the project are

listed. Also, the analytical results for method blanks run during the analysis are given. Results of any matrix spikes, duplicates, matrix spike duplicates, or other project-specific QC are also reported.

Methodology: Reference for analytical methodology used is cited.

12. INTERNAL QC CHECKS

The CCASI QA/QC program continually monitors data quality with internal QC checks. Laboratory Performance QC will determine that laboratory operations are "in control" during data generation. This is based on the use of a standard, controlled matrix to generate precision and accuracy data that is compared, on a daily basis, to control limits. This information, in conjunction with method blank data, is used to assess daily laboratory performance.

Matrix-Specific QC will determine what effect, if any, the sample matrix has on the data being generated. This is based on the use of an actual environmental sample for precision and accuracy determinations, and it commonly relies on the analysis of matrix duplicates, matrix spikes, and matrix spike duplicates. This information, supplemented with field blank and trip blank results, is used to assess the effect of the matrix and field conditions on analytical data.

Laboratory Performance QC is provided as a standard part of every routine CCASI analysis. Matrix-specific QC is available as an option to the client, and it should be specified based on the types of matrices to be analyzed, the

Data Quality Objectives (DQOs), and the regulatory requirements of the project. Matrix-specific QC samples are billable if less than fourteen (14) samples are sent for analysis. Project-specific quotations may include matrix-specific QC samples at no extra charge. Alternate arrangements can be made prior to sample receipt based on project requirements. A complete discussion of the CCASI Internal QC Program follows.

Laboratory Performance QC Program

Laboratory Performance QC is provided as a standard part of every routine CCASI analysis. The main elements of Laboratory Performance QC are:

- The analysis of Laboratory Control Samples, Laboratory Control Sample Duplicates, and Method Blanks; and
- The generation of daily calibration data.

Laboratory Control Samples (LCS) and Laboratory Control Sample Duplicates (LCSD)

LCSs and LCSDs are used to monitor the precision and accuracy of the analytical system on an on-going basis. Each LCS and LCSD consists of a standard, controlled matrix that is spiked with a group of target compounds representative of the method analytes. An LCS pair (consisting of one LCS and one LCSD

spiked at the same levels) is analyzed with each batch of a particular matrix in sets of less than or equal to twenty (20). Client projects which contain less than ten (10) samples are batched together in order to provide an LCS Pair. In the event of a project with less than ten (10) samples in which batching is not an option, the LCS sample only is permitted and deemed adequate. A separate LCS pair is processed for each method being performed on the samples. These are analyzed along with environmental samples to provide evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

Accuracy (average recovery of each analyte in the LCS pair) and precision (Relative Percent Difference [RPD] between each analyte in the LCS pair) data is compared to control limits that have been established for each of the analytes contained in the LCS pair. Initially, control limits for analytes spiked into the LCS are taken directly from methods outlined in SW-846 (see reference in Section 10 of this document). If none are available here, they are taken from the CLP program. As sufficient laboratory data becomes available, the control limits are redefined based upon the most recent ten sets of LCS/LCSD data. Control limits for accuracy for each analyte

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are based on the historical average recovery (mean of the average recoveries of the LCS pairs) plus or minus three standard deviation units. Control limits for precision for each analyte range from zero (no difference between LCS results) to thirty (30) percent.

Analytical data that is generated with an LCS pair which falls within the established control limits is judged to be "in control." Data generated with an LCS pair which falls outside of the control limits is considered suspect, and the analysis is repeated, or results are reported with qualifiers. However, if LCS/LCSD results fall outside of redefined QC limits but are still within original method QC limits, the data will not be deemed unacceptable. If data is determined to be unacceptable, steps must be taken in order to establish validity of the data. This includes examination of instrument performance, preparation and analysis information, consultation with the Group Leader, and finally, a decision as to whether re-analysis is warranted.

LCSs/LCSDs have been established for each routine analytical method. Reagent water is used as a control matrix for the analysis of liquid samples. The LCS compounds are spiked into reagent water and carried through the appropriate steps

of the analysis. The control matrix for solid samples is standard Ottawa sand or anhydrous sodium sulfate. The LCS compounds are spiked into the Ottawa sand or sodium sulfate and carried through the appropriate steps of the analysis.

Method Blanks

Method Blanks, also known as reagent, analytical, or preparation blanks, are analyzed to assess the level of background interference or contamination which exists in the analytical system and which might lead to the reporting of elevated concentration levels or false positive data. As part of the standard CCASI QC program, a method blank is analyzed with every batch of samples processed. A method blank consists of reagents specific to the method which are carried through every aspect of the procedure, including preparation, clean-up, and analysis. The results of the method blank analysis are evaluated, in conjunction with other QC information, to determine the acceptability of the data generated for that batch of samples.

Ideally, the concentration of target analytes in the blank should be below the Reporting Limit for that analyte. In practice, however, some common laboratory solvents and metals

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are difficult to eliminate at ..ne parts-per-billion levels typically reported in environmental analyses. Therefore, criteria for determining blank acceptability must be based on consideration of the analytical techniques used, analytes reported, and Reporting Limits required.

For organic analyses, it is CCASI's policy that the concentration of target analytes in the blank must be below the Reporting Limit for that analyte in order for the blank to be considered acceptable. An exception is made for common laboratory contaminants (methylene chloride, acetone, 2-butanone, toluene, and bis-2-ethylhexyl phthalate) which may be present in the blank at up to 5 times the Reporting Limit and still be considered acceptable. Due to the sensitivity of high resolution dioxin/furan analysis, it is almost completely impossible to eliminate octachlorodibenzo-p-dioxin and furan contamination at part-per-quadrillion levels. This policy is consistent with EPA CLP policy and has been established in recognition of the fact that these compounds are frequently found at low levels in method blanks due to the material used in the collection, sample preparation, and analysis of samples for organic parameters.

For metals analyses, where the Reporting Limits are typically

near the Instrument Detection Limits (IDLs), and background levels for certain metals are difficult to completely eliminate, CCASI's policy is that the concentration of the target analytes in the blank must be below two times the Reporting Limit. If the blank value for a target analyte lies below the Reporting Limit, the Reporting Limit for that analyte in the associated samples is unaffected. If the blank value lies between the Reporting Limit and two times the Reporting Limit, the Reporting Limit for that analyte in the associated samples is raised to the level found in the blank. A blank containing an analyte above two times the Reporting Limit is considered unacceptable unless the lowest concentration of the analyte, in the associated samples, is at least ten times the blank concentration. This is consistent with established EPA CLP policy.

For conventional inorganic tests, the method SOP dictates how the blank is to be treated. Generally, a reagent blank is used both to zero the equipment and as one of the calibration standards. If a preparation step is required for the analysis, then a preparation blank is also analyzed to determine the extent of contamination or background interference. In most cases, the concentration found in the preparation blank is not subtracted from the concentration

found in any associated sample prior to calculating the final result. Blanks have no application or significance for some conventional inorganic parameters (i.e. pH).

If the blank does not meet acceptance criteria, the source of contamination must be investigated, and appropriate corrective action must be taken and documented. Investigation includes an evaluation of the data to determine the extent and effect of the contamination on the sample results. Corrective actions may include re-analysis of the blank, and/or repreparation and re-analysis of the blank and all associated samples.

For conventional organic and metals analyses, and selected conventional inorganic tests, method blank results are reported with each set of sample results. Samples are <u>not</u> corrected for blank contamination.

<u>Matrix-Specific QC</u>

Matrix-Specific QC is used to assess the effects of a sample matrix or field conditions on the analytical data. The main elements of Matrix-Specific QC are:

- The analysis of matrix duplicates, matrix spikes, and matrix spike duplicates;
- Monitoring the recovery of surrogate compounds from

environmental samples;

- Monitoring the results of standard additions in environmental samples; and
- The analysis of field blanks.

Different regulatory programs have different requirements in terms of Matrix-Specific QC. In order to ensure that the data generated meet all Data Quality Objectives, CCASI encourages its clients to include Matrix-Specific QC that fulfill the Data Quality Objectives and regulatory requirements of the project. A discussion of the different elements of Matrix-Specific QC follows.

Matrix Duplicates, Matrix Spikes, and Matrix Spike Duplicates

A Matrix Duplicate (MD) is an environmental sample that is divided into two separate aliquots. The aliquots are processed separately, and the results are compared to determine the effects of the matrix on the precision of the analysis. Results are expressed as RPD.

A Matrix Spike (MS) is an environmental sample to which known concentrations of analytes have been added. The MS is taken through the entire analytical procedure, and the recoveries of the analytes are calculated. Results are expressed as percent recovery. The MS is used to evaluate the effect of

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the sample matrix on the accuracy of the analysis.

A Matrix Spike Duplicate (MSD) is also a environmental sample to which known concentrations of analytes have been added. The MSD is taken through the entire analytical procedure, and the recoveries of the analytes are calculated. The MS and MSD are used to evaluate the effect of the matrix on the precision and accuracy of the analysis. Results are expressed as RPD and percent recovery.

Surrogate Recoveries and Standard Additions

Surrogates are organic compounds which are similar to the analytes of interest in chemical behavior, but which are not normally found in environmental samples. Surrogates are added to samples to monitor the effect of the matrix on the accuracy of the analysis. Results are reported in terms of percent recovery.

CCASI routinely adds surrogates to samples requiring GC/MS and GC/MS/MS analysis and reports these surrogate recoveries to the client. The laboratory does not control its operations based on surrogate recoveries in environmental samples. As discussed earlier in this section, CCASI controls its operations based on the results of Laboratory

Control Samples. The surrogate recoveries are primarily used by the laboratory to assess matrix effects. However, obvious problems with sample preparation and analysis (i.e. evaporation to dryness, leaking septum, etc.) which can lead to poor surrogate spike recoveries must be ruled out prior to attributing low surrogate recoveries to matrix effects.

Standard Additions (SA) is the practice of adding a series of known amounts of an analyte to an environmental sample. The fortified samples are then analyzed, and the recoveries of the analytes are calculated. The practice of SA is generally used with metal and conventional analyses to determine the effect of the sample matrix on the accuracy of the analyses.

Field Blanks

Field blanks are check samples that monitor contamination originating from the collection, transport, or storage of environmental samples. One example of a field blank is an equipment blank. An equipment blank is blank water that is poured through the sample collection device to check the adequacy of the cleaning procedures for the sampling equipment. Another type of field blank is a trip blank. A trip blank is a laboratory control matrix (typically water) which is sent to the field in an appropriate sample

container, remains unopened in the field, and then is sent back to the laboratory. The purpose of the trip blank is to assess the impact of field and shipping conditions on the samples. The results from field blanks are reported to the client as samples in the same concentration units as the samples. No correction of the analytical data is done in the laboratory based on the analysis of field blanks.

13. PERFORMANCE AND SYSTEM AUDITS

A Performance Audit verifies the ability of the laboratory to correctly identify and quantitate compounds in blind check samples submitted by an auditing agency. They are conducted at CCASI through our participation in external quality control programs. Performance Evaluation samples from EPA, other federal/state agencies, or commercial client programs are received, analyzed, verified, and results reported as these studies are conducted. The purpose of these audits is to identify those laboratories that are capable of generating scientifically sound data.

A System Audit is a review of laboratory operations conducted to verify that the laboratory has the necessary facilities, equipment, staff, and procedures in place to generate acceptable data. Systems and performance audits, as an evaluation of all components of the measurement system, are performed by state, federal, and private agencies as part of CCASI participation in sample analysis for both private and governmental organizations.

In addition to external audits conducted by certifying or accrediting agencies or clients, CCASI regularly conducts

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internal calendar quarterly system audits. Internal performance checks are done through the routine analysis of Laboratory Control Samples as outlined in Section 12 of this document.

14. PREVENTIVE MAINTENANCE

To minimize downtime and interruption of analytical work, preventive maintenance is routinely performed on each analytical instrument. All analysts are trained in routine maintenance procedures for all major instrumentation. When repairs are necessary, they are performed by the analyst or trained service engineers employed by the instrument manufacturer.

Each CCASI department has detailed preventive maintenance procedures and schedules on file. Each department is also responsible for maintaining a logbook detailing preventive maintenance and repairs performed on each analytical instrument.

In addition, service contracts are held with the manufacturers of most of the major instrumentation located at CCASI. This ensures prompt, technical service in the event that preventive maintenance uncovers more extensive problems, or other problems arise unexpectedly.

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15. SPECIFIC ROUTINE PROCEDURES USED TO ASSESS DATA QUALITY

Data Quality Assessment

The effectiveness of a QA program is measured by the quality of data generated in the laboratory. Data quality is judged in terms of its precision, accuracy, representativeness, completeness, and comparability. These terms are described as follows:

Precision is the degree to which the measurement is reproducible. Precision can be assessed by replicate measurements of laboratory spikes, reference materials, or environmental samples. Precision is routinely monitored by comparing the RPD between LCS pair measurements with control limits established at plus or minus three standard deviation units from the mean RPD of historical LCS data. The standard deviation of "n" measurements of "x" is commonly used to estimate precision.

Standard deviation (s) is calculated as follows:

$$s = \sqrt{\frac{1}{n-1} \sum_{i=1}^{n} (X - \bar{X})^2}$$

where a quantity "x" (i.e. a concentration) is measured "n"

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times and $X = \sum_{i=1}^{n} \frac{X}{n}$.

The relative standard deviation, which expresses standard deviation as a percentage of the mean, is generally useful in the comparison of three or more replicates.

$$RSD = 100\ (s/\bar{x})$$

where: RSD = relative standard deviation
s = standard deviation
x = mean

In the case of duplicates, the RPD between the two samples may be used to estimate precision.

$$RPD = \frac{|D_1 - D_2|}{(D_1 + D_2)/2} \times 100$$

where: RPD = relative percent difference
D = first sample value
D = second sample value (duplicate)

Accuracy is a determination of how close the measurement is to the true value. Accuracy can be assessed using LCS results, standard reference materials, or spiked environmental sample results. Accuracy is monitored by comparing LCS results with true analyte concentrations.

The determination of the accuracy of a measurement requires a knowledge of the true or accepted value for the analyte being

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measured. Accuracy may be calculated in terms of percent recovery as follows:

$$\text{Percent Recovery} = \left(\frac{X}{T}\right) \times 100$$

where X = the observed value of measurement
T = "true" value

Control limits are established at plus or minus three standard deviation units from the mean historical percent recovery results.

Representativeness is the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition. Analytical data should represent the sample analyzed, however, the heterogeneity of the original sample matrix can have an adverse effect on the result.

Completeness is a measure of the amount of valid data obtained from a measurement system compared with the amount that was expected to be obtained under normal conditions. To be considered complete, the data set must contain all QC check analyses verifying precision and accuracy for the analytical protocol.

When possible, the percent completeness for each set of samples is calculated as follows:

$$\text{Completeness} = \frac{\text{Valid data obtained}}{\text{Total data planned}} \times 100\%$$

Comparability expresses the confidence with which one data set can be compared to another data set measuring the same property. Comparability is ensured through the use of established and approved analytical methods, consistency on the basis of analysis (i.e. wet weight, volume, etc.), consistency in reporting units (i.e. ppm, ppb, etc.), and analysis of standard reference materials.

16. CORRECTIVE ACTION

When errors, deficiencies, or out-of-control situations occur, the QA program provides systematic procedures called "corrective actions" to resolve problems and restore proper functioning to the analytical system.

Laboratory personnel are alerted that corrective actions may be necessary if:

- QC data are outside the acceptable windows for precision and accuracy; or
- Blanks or LCS contain contaminants above acceptable levels; or
- Undesirable trends are detected in spike recoveries or RPD between duplicates; or
- Deficiencies are detected by the QA/QC Officer during internal or external audits or from the results of performance evaluation samples; or
- Inquiries concerning the data are received from clients.

Corrective action procedures are often handled at the bench level by the analyst. He/She reviews the preparation or extraction procedure for possible errors, checks the instrument calibration, spike and calibration mixes, instrument sensitivity, etc. If the problem persists or cannot be identified, the matter is referred to the Operations Manager and/or QA/QC Officer for further

investigation. Once resolved, full documentation of the corrective action procedure is filed with the QA/QC Officer and with all project files that were affected.

Follow-up will also be done by the QA/QC Officer and/or the Operations Manager to ensure that the corrective action was properly implemented and that the errors, deficiencies, or out-of-control situations have been adequately corrected. Documentation of this follow-up is also filed with the QA/QC Officer and with all project files that are affected.

Nonconformances that occur (i.e. samples received without proper documentation or in improper containers) are also documented and filed in the same manner as corrective actions. If a nonconformance repeatedly occurs, corrective action procedures are initiated as described above.

17. QA REPORTS TO MANAGEMENT

The reporting system is a valuable tool for measuring the overall effectiveness of the QA program. It serves as an instrument for evaluating the program design, identifying problems and trends, and planning for future needs. The following items are reported to management at the time of occurrence or the completion of the event:

- The results of internal systems audits including any corrective action taken;
- Performance evaluation scores and commentaries;
- Results of site visits and audits by regulatory agencies and clients;
- Performance on major contracts;
- Problems encountered and corrective actions taken;
- Holding time violations;
- Comments and recommendations; and
- A summary of the QA data audits conducted.

Meetings are held between the QA/QC staff and other management staff on a weekly basis.

18. LABORATORY DOCUMENTATION

Complete and accurate documentation of analytical and procedural information is an important part of the QA Program. The following discussion addresses the different types of documentation used at CCASI.

SOPs

Details of analytical and QC protocols are contained in SOPs. SOPs are documents that contain detailed information on the requirements for the performance of a laboratory procedure. All SOPs are approved by the QA/QC Officer before implementation.

Laboratory Bench Sheets

Laboratory bench sheets are used to document information from routine laboratory operations including sample preparation and analysis. Bench sheets are used to ensure that the information is recorded in a complete and organized manner, and that the analysis can be reconstructed if necessary.

Laboratory Notebooks

Laboratory notebooks are used to document all information

relating to sample preparation and analysis. Information typically recorded in laboratory notebooks includes unusual observations or occurrences in the analysis of samples or in methods development. Each page in a laboratory notebook is initialed and dated as information is entered.

Project Files

A project file is created for each project handled within the laboratory. The project file contains all documents associated with the particular project. This includes correspondence from the client, telephone conversation logs, chain-of-custody records, raw data, laboratory bench sheets, copies of laboratory notebook entries pertaining to the project, and a copy of the final report. When a project is complete, all records are passed to the Document Control Officer who inventories the file, checks for completeness, distributes final report to client, and places the file into document archives.

APPENDIX I

MAXIMUM HOLDING TIMES AND SAMPLE COLLECTION/PERSERVATION INFORMATION

Sources: SW-846, 3rd Edition, Update I
40 CFR, part 136, July 1, 1991

(CCAS Indianapolis QA Program Plan, Revision 3.0)

Table 1

VOLATILE ORGANICS

Matrix	Container	Minimum Sample Size	Preservative	Holding Time (From Date Sampled)
Water Samples				
No Residual Chlorine Present	Two 40 mL vials with Teflon lined septum caps	40 mL	0.5 mL 1:1 HCL, 4°C	14 days
Residual Chlorine Present	Two 40 mL vials with Teflon lined septum caps	40 mL	0.5 mL of 10% sodium thiosulfate, 0.5 mL 1:1 HCL, 4°C	14 days
Soil/Sediments and Sludges	Glass jar with Teflon liner	10 g	4°C	14 days
Concentrated Waste Samples	Glass jar with Teflon liner	10 g	None	14 days

The above information applies to the following parameters and methods:

Parameter	Method
Volatile Halocarbons	502.2/601/8010 (GC)
Volatile Aromatics	502.2/503.1/602/8020 (GC)
Volatile Organics	524.2/624/8240 (GC/MS)
	502.2 (GC)
Trihalomethanes	501.1 (GC)
EDB/DBCP	504 (GC)

Table 2

SEMIVOLATILE ORGANICS

Matrix	Container	Minimum Sample Size	Preservative	Holding Time (From Date Sampled)
Water Samples				
No Residual Chlorine Present	1 liter glass amber with Teflon liner	1 liter	4° C	Samples must be extracted within 7 days and analyzed within 40 days of extraction
Residual Chlorine Present	1 liter glass amber with Teflon liner	1 liter	Add 3 mL 10% sodium thiosulfate per gallon, 4° C	Samples must be extracted within 7 days and analyzed within 40 days of extraction
Soil/Sediments and Sludges	Glass jar with Teflon liner	50 g	4° C	Samples must be extracted within 14 days and analyzed within 40 days of extraction
Concentrated Waste Samples	Glass jar with Teflon liner	50 g	None	Samples must be extracted within 14 days and analyzed within 40 days of extraction

The above information applies to the following parameters and methods:

Parameter	Method
Phenols	604/8040 (GC)
Organochlorine Pesticides/PCBs	505/508/508A/608/8080 (GC)
Polynuclear Aromatic Hydrocarbons	550/610/8310 (HPLC)
Phenoxy acid Herbicides	615/8150 (GC)
Semivolatile Organics	625/8270 (GC/MS)
Carbamate Pesticides	531.1/632 (HPLC)

Table 3

OTHER ORGANICS

Parameter	Method Nos.	Matrix	Holding Time (a) (from Date Sampled)	Container	Preservative
Dioxins/Furans	8280; DFLM01.1 (CLP SOW)	Water	None	1 liter glass amber w/Teflon	4 °C
		Soil/Waste	None	Glass jar w/ Teflon liner	None
Dioxins/Furans	1613	Water	None 40 days anal.(b)	1 liter glass amber w/Teflon	80 mg/l sodium thiosulfate, 4 °C
		Soil/Waste	None 40 days anal.(b)	Glass jar w/ Teflon liner	None
2,3,7,8-TCDD	11/92 SOW (Rapid Turnaround)	Water	None	1 liter glass amber w/Teflon	None
		Soil/Waste	None	Glass jar w/ Teflon liner	None
Glyphosate	547	Water	14 days	Two 40 mL amber vials w/Teflon lined septum caps	100 mg/l sodium thiosulfate, 4 °C
Endothall	548	Water	7 days 1 day anal. (b)	Two 40 mL amber vials w/Teflon lined septum caps	4 °C
Diquat/Paraquat	549	Water	7 days 21 days anal. (b)	1 liter PVC, high density	H2SO4 to pH<2 4 °C

(a) extn: extraction anal: analysis
(b) from date of extraction

Table 4 OTHER ORGANICS (cont.)

Parameter	Method Nos.	Matrix	Holding Time (a) (from Date Sampled)	Container	Preservative
Petroleum Hydrocarbon Fingerprint	TPH-Fingerprint Purge & Trap Mod. 8015	Water	14 days	2 40 mL vials w/Teflon liners	4°C, HCL to pH < 2
		Soil/Waste	14 days	Glass jar w/ Teflon liner	4°C
Petroleum Hydrocarbons (TPH)	TPH-IR (418.1)	Water	28 days	1 liter glass w/Teflon liner	4°C, H2SO4 to pH < 2
		Soil/Waste	28 days	Glass jar w/ Teflon liner	4°C

(a) extn: extraction anal: analysis
(b) from date of extraction

Table 5

METALS

Parameter	Method Nos.	Matrix	Holding Time (From Date Sampled)	Container	Preservative (a)	Min. Sample Size
Metals (ICP)	200.7/6010	Water Soil/Waste	6 months 6 months	Poly Glass jar w/ Teflon liner	HNO3 to pH<2.0 4°C	100 ml 10 g
Arsenic (GFAA)	206.2/7060	Water Soil/Waste	6 months 6 months	Poly Glass jar w/ Teflon liner	HNO3 to pH<2.0 4°C	100 ml 10 g
Mercury (CVAA)	245.1/7470	Water Soil/Waste	28 days 28 days	Poly Glass jar w/ Teflon liner	HNO3 to pH<2.0 4°C	100 ml 10 g
Selenium (GFAA)	270.2/7740	Water Soil/Waste	6 months 6 months	Poly Glass jar w/ Teflon liner	HNO3 to pH<2.0 4°C	100 ml 10 g
Thallium (GFAA)	279.2/7841	Water Soil/Waste	6 months 6 months	Poly Glass jar w/ Teflon liner	HNO3 to pH<2.0 4°C	100 ml 10 g
Lead (GFAA)	239.2/7421	Water Soil/Waste	6 months 6 months	Poly Glass jar w/ Teflon liner	HNO3 to pH<2.0 4°C	100 ml 10 g

(a) Listed preservative is for total metals. Dissolved or suspended metals require filtration prior to pH adjustment.

Table 6 CONVENTIONAL INORGANICS

Parameter	Method Nos.	Matrix	Holding Time (from Date Sampled)	Container	Preservative	Min. Sample Size
Acidity	305.1	Water	14 days	Poly	4°C	100 ml
Alkalinity	310.1	Water	14 days	Poly	4°C	100 ml
Ammonia	350.3	Water Soil/Waste	28 days 28 days	Poly Glass jar w/ Teflon liner	H_2SO_4 to pH<2 4°C	400 ml 10 g
Chloride	Orion Manual	Water Soil/Waste	28 days 28 days	Poly Glass jar w/ Teflon liner	4°C 4°C	50 ml 10 g
Cyanide, Total	335.3/9010	Water Soil/Waste	14 days 14 days	Poly Glass jar w/ Teflon liner	NaOH to pH>12 4°C	1000 ml 10 g
Fluoride	340.2	Water Soil/Waste	28 days 28 days	Poly Glass jar w/ Teflon liner	4°C 4°C	100 ml 10 g
Chromium IV	7196	Water	hrs.	Poly	4	200 ml
Nitrate	352.1	Water Soil/Waste	48 hrs. 14 days 48 hrs.	Poly Glass jar w/ Teflon liner	4°C H_2SO_4 to pH<2.0 4°C	100 ml 100 ml 10 g
Nitrite	4500-NO3-D	Water Soil/Waste	48 hrs. 48 hrs.	Poly Glass jar w/ Teflon liner	4°C 4°C	50 ml 10 g
Oil and Grease	431.1/9070	Water Soil/Waste	28 days 28 days	Glass jar w/ Teflon liner (both matrices)	H_2SO_4 to pH<2.0 4°C	1000 ml 10 g

Table 7

CONVENTIONAL INORGANICS (cont.)

Parameter	Method Nos.	Matrix	Holding Time (from Date Sampled)	Container	Preservative	Min. Sample Size
Orthophosphate	365.3	Water Soil/Waste	48 hrs. 48 hrs.	Poly Glass jar w/ Teflon liner	Filter Immed., 4°C 4°C	50 ml 10 g
Phosphorus, Tot.	365.3	Water Soil/Waste	28 days 28 days	Poly Glass jar w/ Teflon liner	H_2SO_4 tp pH<2.0 4°C	50 ml 10 g
Residue, Total	160.3	Water Soil/Waste	7 days 7 days	Poly Glass jar w/ Teflon liner	4°C 4°C	100 ml 10 g
Residue, Filterable (TDS)	160.1	Water	7 days	Poly	4°C	100 ml
Residue, Nonfilterable (TSS)	160.2	Water	7 days	Poly	4°C	100 ml
Residue, Settleable (SS)	160.5	Water	48 hrs.	Poly	4°C	1000 ml
Residue, Volatile (TVS)	160.4	Water	7 days	Poly	4°C	100 ml
Specific Conductance	120.1	Water	28 days	Poly	4°C	100 ml
Turbidity	180.1	Water	48 hrs.	Poly	4°C	250 ml

Table 8 CLP PARAMETERS

Parameter	Matrix	Holding Time (a) (from Date received)	Container	Preservative	Min. Sample Size
Volatile Organics	Water	10 days	Two 40 ml voals with Teflon lined caps	4°C	50 ml
	Soil	10 days	Glass jar with Teflon liner	4°C	10 g
Extractable Organics	Water	5 days extn. 40 days anal.	1 liter amber glass with Teflon liner	4°C	1000 ml
	Soil	10 days extn. 40 days anal.	Glass jar with Teflon liner	4°C	50 g
Metals (other than Mercury)	Water	180 days	P, G (b)	HNO_3 to pH<2.0	100 ml
	Soil	180 days	P, G	4°C	10 g
Mercury	Water	26 days	P, G	HNO_3 to pH<2.0	100 ml
	Soil	26 days	P, G	4°C	10 g
Cyanide	Water	14 days	P, G	$NaOH$ to pH>12.0	100 ml
	Soil	14 days	P, G	4°C	10 g

(a) Holding times calculated from date of receipt in laboratory
(b) Polyethylene (P) or glass (G)

APPENDIX II

SAMPLE COLLECTION GUIDELINES

(CCAS Indianapolis QA Program Plan, Revision 3.0)

SAMPLE COLLECTION GUIDELINES

1. The containers that have been supplied to you are those that are appropriate for the parameters for which you are sampling. **Please fill all the containers!!**

2. If you are collecting water samples that require preservation, the proper preservatives have been added to the containers. **HANDLE WITH CARE!!** We recommend that you wear gloves during sample collection. Also, try to avoid rinsing the preservative out of the container.

3. For any matrix being collected, containers should be filled as much as possible. If collecting samples for volatile organics, do not leave any headspace in the container.

4. Please fully complete and enclose chain-of-custody documentation. Samples cannot be efficiently processed without this information.

5. All samples must be stored and shipped at 4°C. If blue ice packs are not available, please use ice that is in sealed plastic bags. Also, to avoid jeopardizing sample integrity, we recommend that all samples be placed in sealed plastic bags or individually wrapped in plastic shipping material.

6. Coolers should be well sealed and shipped using an overnight carrier. This will help to insure that samples remain completely cooled during shipping.

7. If questions should arise during the sample collection process, please contact our Client Services Representative at 1-800-875-8674.

pace
INCORPORATED
ENVIRONMENTAL LABORATORIES

QUALITY ASSURANCE PLAN

OF

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INTRODUCTION/STATEMENT OF POLICY

This is a generic Quality Assurance Plan (QAP) designed to give an overview of the organization,structure, analytical capabilities and services, and the quality assurance/quality control (QA/QC) program used by PACE, Inc. The QA objectives of the program are to control, assess, and document data quality. The program achieves these objectives through two distinct, interrelated functions: (1) providing quality control data that can be used to determine precision and accuracy, and (2) controlling data quality within acceptable limits.

This manual identifies laboratory methods published by the U.S. Environmental Protection Agency and other authorities, and describes the quality control procedures to be used with them, but is not intended to serve as a manual of standard operating procedures. All current Standard Operating Procedures (SOPs) are kept on file at the laboratory. As they are needed, these more detailed procedures can be provided on a project-specific basis.

As stated this is a generic QAP and is not intended to be a Quality Assurance Project Plan specific to the client's requirements and requests for a given sample work site. If a detailed QAPP is desired or needed, we will work closely with the client to develop one.

Note that this is a confidential laboratory controlled document. This document is not to be edited, taken apart, or distributed without the explicit consent of this laboratory. Your cooperation is appreciated.

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SECTION 3

STATEMENT OF POLICY

This section currently is incorporated into the introduction. This section will be independent in order to comply with state QAP formats.

SECTION 4

LABORATORY ORGANIZATION AND CAPABILITIES

PACE, Inc. is a national system of laboratories that provide environmental analytical testing services for commercial and government clients.

4.1 Organization

PACE, Inc. is composed of nine functional groups. Each functional group is headed by a department manager who is responsible for the group's technical performance. Department managers and their respective groups are described in the organization chart provided in Table 4.0. These functional groups are defined as follows:

Sample Management
Client Services
Organic Sample Preparation (Organic Prep)
Gas Chromatography (GC)
Gas Chromatography/Mass Spectrometry (GC/MS)
Trace Metals Analysis (Metals)
Conventionals (Water Quality)
Report Center
Quality Assurance/Quality Control (QA/QC)

4.11 Sample Management

The Sample Management department is responsible for the receipt, inspection and verification of sample containers, log-in of samples into the Laboratory Information Management System (LIMS), and sample distribution to their proper storage environments through out the laboratory. Sample Management also ensures the client is supplied with the necessary bottles with consistent and accurate sample preservation. If sample aliquoting is necessary, Sample Management will perform the aliquoting, labeling, and distributing for the entire laboratory. Upon completion of analysis and the expiration of contract specified sample or extract hold time, Sample Management will perform the correct method of sample disposal.

4.12 Client Services

The Client Services department is the primary contact for communications with the client. The individuals manage each project from the initiation of quotes and proposals until the report is mailed. The representatives ensure that all project requirements and requests are communicated to the appropriate laboratory personnel. Non-conformances and/or problems encountered during analysis are addressed to the client through the Client Service's representative.

4.13 Organic Sample Preparation

The Organic Sample Preparation (Organic Prep) department is responsible for method preparation of all samples for organic analyses, except for volatile organics which require little or no sample preparation. If volatile analysis sample preparation is necessary, it is preformed by the GC or GC/MS analyst who is performing the analytical testing. Sample cleanup and screening is performed by the Organic Prep group when necessary. The Organic Prep group ensures that sample integrity is maintained while being prepared. It is the responsibility of the Organic Prep manager to ensure samples are extracted within the specified method hold times as well as that samples extractions are performed in accordance with method specifications.

An extraction log is given to the appropriate analytical laboratory upon completion of a sample batch. Extracts are stored in their corresponding refrigerators.

4.14 Gas Chromatography

Two separate analytical laboratories exist within the GC department. The GC manager oversees both the Volatiles (VOA) and Extractables laboratories. It is the responsibility of the GC analyst to maintain the proper operation of the instrument and ensure the instrument is calibrated in accordance with method criteria. The GC analyst is also responsible for maintaining the integrity of the sample (VOAs) or the extract during the analytical run. The analyst gathers and consolidates the analytical data produced for each sample into a data package. This data package is reviewed by the GC manager. It is the manager's responsibility to ensure that all method and QA/QC specifications are met and documented. The reviewed data package is submitted to and registered with the Report Center.

4.15 Gas Chromatography/Mass Spectrometry

Two separate analytical laboratories exist within the GC/MS department. The GC/MS manager oversees both the Volatiles (VOA) and Extractables laboratories. It is the responsibility of the GC/MS analyst to maintain the proper operation of the instrument and ensure the instrument is calibrated in accordance to method criteria. The analyst is responsible for maintaining the integrity of the sample (VOAs) or the extract during the analytical run. The analyst gathers and consolidates the analytical data produced for each sample into a data package. This data package is reviewed by the GC/MS manager/supervisor. It is the manager's responsibility to ensure that all method and QA/QC specifications are met and documented. The reviewed data package is submitted to and registered with the Report Center.

4.16 Trace Metals Analysis

The Trace Metals Analysis (Metals) department is responsible for the preparing and analyzing of all samples requiring trace metal analyses. The Metals analyst is responsible for maintaining sample integrity through out sample preparation and analysis. The analyst must perform any maintenance necessary to ensure the proper operation of the instrument. The analyst is also responsible for seeing that the instrument is calibrated according to method guidelines. The analyst gathers and condenses the analytical data collected for each sample into a data package. This data package is thoroughly review by the Metals manager/supervisor. The manager is responsible for ensuring that all QA/QC method requirements are met. The data package is submitted to and registered with the Report Center.

4.17 Conventionals

The Conventionals (Water Quality) department encompasses general wet chemistry or biological parameters. The Water Quality analyst is responsible for making all necessary reagents, setting up of distillation (or any other) apparatus needed, and maintaining proper instrument operation for the analytical testing. The analyst is responsible for the calibrating of the instrument and/or standardization of titration reagents. The analyst records and condenses all analytical data. The Water Quality manager ensures that all QA/QC requirements are fulfilled. The manager reviews the analytical bench sheets and generates a data package which is submitted and registered with the Report Center.

4.18 Report Center

The Report Center is responsible for the issuance and revisions of reports to clients in a timely manner and in the appropriate report format. The Report Center generates all final reports submitted to the client. The staff's function is to review reports generated from the department data packages and correspond with the laboratory manager if problems or discrepancies occur. The Report Center will track the progress of data packages within the lab. The Report Center manager is responsible for the tracking and archival of all reports.

4.19 Quality Assurance

The Quality Assurance (QA) office provides an independent laboratory check that quality is maintained through out the laboratory. The QA office is responsible for the scheduling and conducting of routine internal audits and in house performance quality control (QC) checks. The QA office spot reviews complete report data packages before and after issuance to the client. The QA office initiates and monitors the QA log process and issues QA memorandums when a systematic or procedural error is discovered. The QA office maintains records of all MDL and QC studies performed throughout the laboratory. Department SOPs must be approved by the QA office, and the QA office authorizes any revisions or replacements of current SOPs. The QA office approves all report resubmissions before their release.

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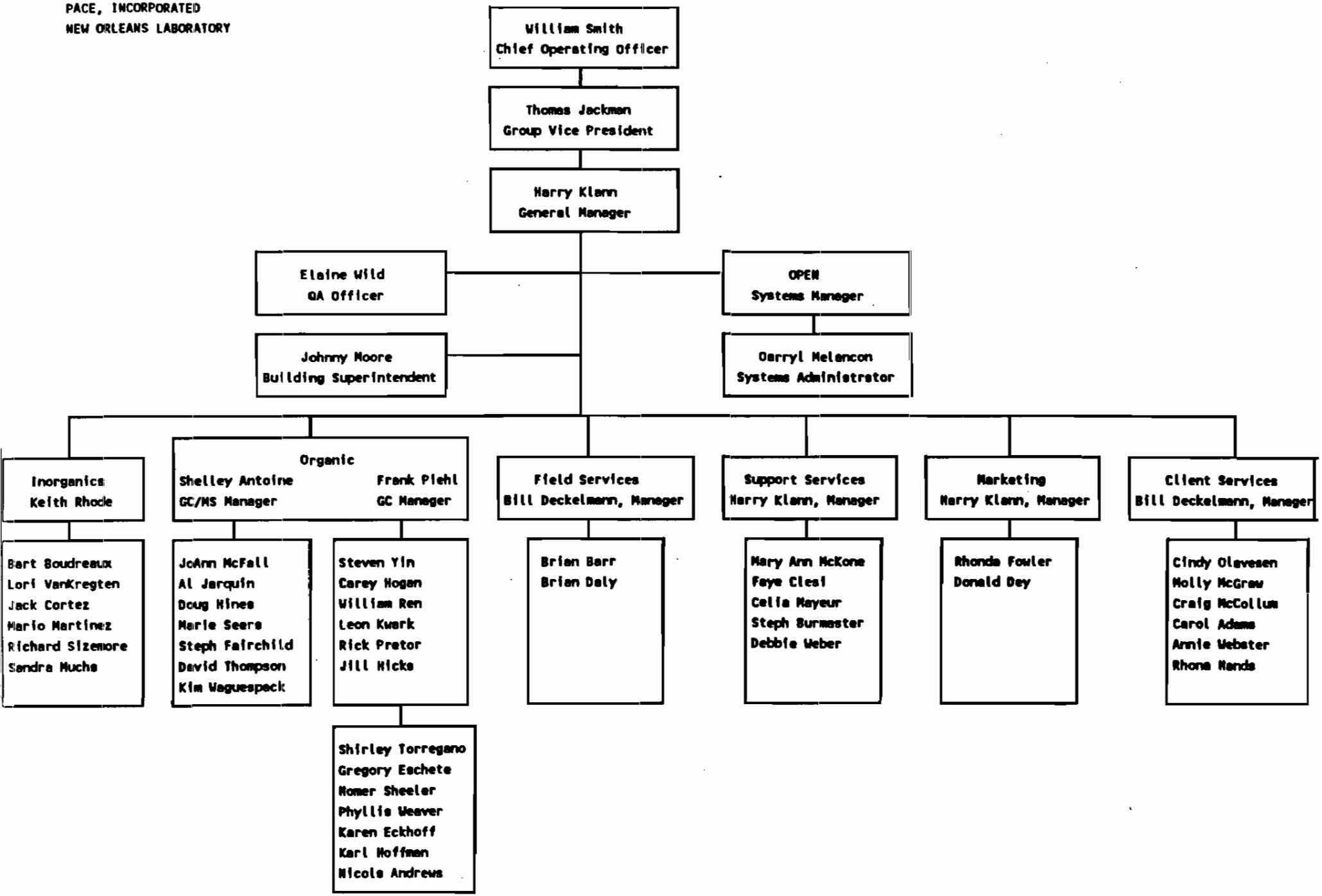


4.1 Analytical Services

PACE, Inc. provides a full range of environmental testing for organic and inorganic constituents in a variety of matrices found in environmental and hazardous waste samples. These services range from commonly requested analyses to site-specific analyses. Our experienced staff also can assist clients by providing methods development, method validation, data management, and data interpretation. The following is a list of the more routinely performed services. To further define these services Tables 4.1 through 4.5 list the preparation and analytical methods routinely performed by the laboratory.

COMPREHENSIVE ENVIRONMENTAL RESPONSE COMPENSATION AND LIABILITY ACT/SUPERFUND AMENDMENTS AND REAUTHORIZATION ACT (CERCLA/SARA)

- Contract Laboratory Program (CLP) Participant
  Seven years' continuous program participation
- Routine Analytical Services (RAS)
- Special Analytical Services (SAS)
- High Concentration Sample Program, Methods Development, and Analysis
- Inorganics, Target Analyte List (TAL)
- Organics, Target Compound List (TCL)
- SARA Title III

NATIONAL POLLUTANT DISCHARGE ELIMINATION SYSTEM (NPDES)

- Form 2C Permit Support
  - Required Pollutants
  - Priority Pollutants
  - Conventional Parameters
  - Nonconventional Parameters
- Priority Pollutants

RESOURCE CONSERVATION AND RECOVERY ACT (RCRA)

- Groundwater Monitoring
  - Suitability Parameters
  - Quality Parameters
  - Contamination Parameters
  - Appendix IX Constituents
- Hazardous Waste Characteristics/Land Disposal Restrictions
  - Ignitability
  - Corrosivity
  - Reactivity
  - EP Toxicity
  - Toxic Characteristic Leaching Procedure (TCLP)
  - California List
  - Petroleum Refinery Waste List (Skinner List)
  - Appendix III
  - Appendix VII
  - Appendix VIII

- Underground Storage Tanks (UST)
  Benzene, Toluene, Ethylbenzene and Xylene (BTEX)
  Total Petroleum Hydrocarbons (TPH)
  Flashpoint
  Solvents Analysis

- Rapid Turnaround

DRINKING WATER

- Primary and Secondary Drinking Water Requirements

- Low Detection Limits for Volatile Compounds

METHODS DEVELOPMENT. Methods development and validation projects include, but are not limited to, the following:

- Designing analytical approaches to characterize and quantify constituents in complex environmental matrices.

- Intra- and interlaboratory methods evaluation

- High-concentration multiphase sample analysis

DATA MANAGEMENT. To assist clients with data management and reporting requirements, PACE, Inc. provides:

- Diskette and electronic transfer of data

- Customized report formats

Standard Analytical Methods and Procedures

Table 4.1

Sample Preparation Methods

Parameter	Method	Description	Associated Analytical Methods
Liquid Extraction	SW3510	Separatory Funnel Liquid-Liquid Extraction	SW 8080, 8270 418.1, CA-DHS
	SW3520	Continuous Liquid-Liquid Extraction	SW 8080, 8270 418.1, CA-DHS
Solid Extraction	SW3550	Sonication Extraction	418.1, CA-DHS
	SW3540	Soxhlet Extraction	SW 8080, 8270
Organic Wastes	SW3580	Waste Dilution	SW 8080, 8270
Volatiles	SW5030	Purge and Trap	SW 8010, 8015, 8020, 8030, 8240
GPC Clean-up	SW3640	Gel Permeation Cleanup	
Column Clean-up	SW3610	Alumina Column Cleanup	SW 8080, 8270
	SW3620	Florisil Column Cleanup	SW 8080, 8270
Acid Wash	600/4-81-045	Sulfuric Acid Wash	EPA 600/4-81-045 SW 8080 (PCB only)
Acid Digestion	SW3050	Acid Digestion of Sediments, Soils, and Sludges	SW 6010,7041,7421,7471, 7740,7841
	SW3020	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by GFAA	SW 6010,7041,7421,7470, 7740,7841
	SW3010	Acid Digestion of Aqueous Samples and Extracts for Total Metals for Analysis by FLAA or ICP Spectroscopy	SW 6010,7041,7421,7470, 7740,7841
TCLP	SW1311	Toxicity Characteristic Leaching Procedure	40CFR 268

Standard Analytical Methods and Procedures

Table 4.2

Gas Chromatography Methods

Parameter	Method	Description	Associated Analytical Methods
Volatile Organics	SW8010	Halogenated Volatile Organics	EPA 601
	SW8015(mod)	Total Petroleum Hydrocarbons-Purgeables	CA-DHS
	SW8020	Aromatic Volatile Organics	EPA 602
	SW8030	Acrolein, Acrylonitrile, Acetonitrile	EPA 603
Phenols	SW8040	Phenols	EPA 604
Organochlorine Pesticides	SW8080	Organochlorine Pesticides and PCBs	EPA 608
	CLP-SOW	3/90	
PCBs	SW8080	Organochlorine Pesticides and PCBs	EPA 608
	CLP-SOW	3/90	
PCBs in Oil	600/ 4-81-045	PCBs in Oil	
PNA (PAH)	SW8100	Polynuclear Aromatic Hydrocarbons	EPA 610
Chlorinated Hydrocarbons	SW8120	Chlorinated Hydrocarbons	EPA 612
Organophosphorus Pesticides	SW8140	Organophosphorus Pesticides	EPA 614

Standard Analytical Methods and Procedures

Table 4.2

Gas Chromatography Methods

Parameter	Method	Description	Associated Analytical Methods
Herbicides	SW8150	Chlorinated Herbicides	EPA 615
TPH	CA-DHS(mod)	Total Petroleum Hydrocarbons - Extractables by GC-FID	8015 M
	EPA 418.1	Total Petroleum Hydrocarbons by IR	

Standard Analytical Methods and Procedures

Table 4.3

GC/MS Methods

Parameter	Method	Description	Associated Analytical Methods
Volatile Organics	SW8240	Gas Chromatography/Mass Spectrometry Volatile Organics	EPA 624
	CLP-SOW	3/90	
Semivolatile Organics	SW8270	Gas Chromatography/Mass Spectrometry for Semivolatile Organics: Capillary Column Technique	EPA 625
	CLP-SOW	3/90	

Standard Analytical Methods and Procedures

Table 4.4

Metals Analysis Methods

Parameter	Method	Description	Associated Analytical Methods
Aluminum	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Antimony	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
	SW7041	Antimony (Atomic Absorption Furnance Technique)	EPA 204.2
Arsenic	SW7060	Arsenic (Atomic Absorption, Furnance Technique)	EPA 206.2
	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Barium	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Beryllium	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Boron	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Cadmium	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
	SW7131	Cadmium (Atomic Absorption Furnance Technique)	EPA 213.2
Calcium	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7

Standard Analytical Methods and Procedures

Table 4.4

Metals Analysis Methods

Parameter	Method	Description	Associated Analytical Methods
Chromium	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Cobalt	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Copper	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Iron	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Lead	SW7421	Lead (Atomic Absorption, Furnance Technique)	EPA 239.2
	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Magnesium	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Manganese	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Mercury	SW7470	Mercury in Liquid Waste (Manual Cold Vapor Technique)	EPA 245.1
	SW7471	Mercury in Solid or Semisolid Waste (Manual Cold Vapor Technique)	EPA 245.5
Molybdenum	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7

Standard Analytical Methods and Procedures

Table 4.4

Metals Analysis Methods

Parameter	Method	Description	Associated Analytical Methods
Nickel	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Potassium	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Selenium	SW7740	Selenium (Atomic Absorption, Furnance Technique)	EPA 270.2
	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Silver	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Sodium	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Thallium	SW7841	Thallium (Atomic Absorption, Furnance Technique)	EPA 279.2
	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Vanadium	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7
Zinc	SW6010	Inductively Coupled Plasma-Atomic Emission Spectroscopy	EPA 200.7

Standard Analytical Methods and Procedures

Table 4.5

Water Quality Methods

Parameter	Method	Description	Associated Analytical Methods
Acidity	EPA 305.1	Acidity (Titrimetric)	
Alkalinity	EPA 310.1	Alkalinity (Titrimetric (pH 4.5))	
BOD	EPA 405.1	Biological Oxygen Demand (5 day, 20C)	
Bromide	EPA 320.1	Bromide (Titrimetric)	
Chloride	SW9252	Chloride (Colorimetric, Automated Ferricyanide)	EPA 325.1, 352.2
Chlorine	EPA 330.4	Total Residual Chlorine (Titrimetric, DPD-FAS)	
COD	EPA 410.4	Chemical Oxygen Demand (Colorimetric, Automated; Manual)	
Color	EPA 110.2	Color	
Conductance	SW9050	Specific Conductance	EPA 120.1
Cr (+6)	SW7196	Hexavalent Chromium (Colorimetric)	SM312B
Cyanide	SW9010	Total and Amenable Cyanide (Colorimetric, Automated UV)[1]	EPA 335.3
	CLP-SOW SW Sec 7.3.3.2	3/90 Reactive Cyanide	
Fluoride	EPA 340.1	Fluoride (Colorimetric)	

Standard Analytical Methods and Procedures

Table 4.5

Water Quality Methods

Parameter	Method	Description	Associated Analytical Methods
Hardness	EPA 130.2	Total Hardness (as CaCO3) (Titrimetric, EDTA)	
Nitrogen	EPA 350.1	Ammonia (Colorimetric; Automated Phenate)	
	EPA 351.2	Total Kjedahl Nitrogen (Colorimetric; Semi-automated Block Digestor AAII)	
	EPA 352.1	Nitrate[2]	
	EPA 353.2	Nitrate-Nitrite (Colorimetric, Automated Cadmium Reduction)	
	EPA 354.1	Nitrite (Spectrophotometric)	
Oil and Grease	SW9070	Total Recoverable Oil & Grease (Gravimetric Separatory Funnel Extraction)	EPA 413.1
	SW9071	Oil & Grease Extraction Method for Sludge Samples	
pH	SW9040	pH Electrometric Measurement	EPA 150.1
	SW9045	Soil pH	
Phenols	SW9065	Phenolics (Spectrophotometric, Automated 4-AAP with Distillation)	EPA 420.2
Phosphorus	EPA 365.1	Phosphorus, All Forms (Colorimetric; Automated Ascorbic Acid)	
	EPA 365.4	Phosphorus, Total (Colorimetric; Automated, Block Digestor AAII)	

Standard Analytical Methods and Procedures

Table 4.5

Water Quality Methods

Parameter	Method	Description	Associated Analytical Methods
Residue	EPA 160.1	Filterable Residue (Gravimetric, Dried at 180C) TDS	
	EPA 160.2	Non-Filterable Residue (Gravimetric, Dried at 103-105C) TSS	
	EPA 160.3	Total Residue (Gravimetric, Dried at 103-105C)	
Silica	EPA 370.1	Dissolved Silica (Colorimetric)	
Sulfate	SW9038	Sulfate (Colorimetric; Automated Methyl Thymol)	EPA 375.2
Sulfide	SW9030	Sulfide (Titrimetric, Iodide)	EPA 376.1
	SW Sec 7.3.4.1	Reactive Sulfide	
Sulfite	EPA 377.1	Sulfite (Titrimetric)	
TOC	SW9060	Total Organic Carbon	EPA 415.1
TOX	SW9020	Total Organic Halides	
Turbidity	EPA 180.1	Nephelometric	

1 Samples are manually distilled prior to the automated colorimetric analysis.

2 Nitrate is reported as the difference between the Nitrate/Nitrite result and the nitrite specific result.

4.2 Facilities and Equipment

PACE, Inc. is equipped with state-of-the-art instrumentation and application software in order to facilitate sample handling and thus enabling the analyst to dedicate more time to the resolution of technical problems during analytical testing. The following is a general overview of the use and application of instrumentation and software. Table 4.6 gives a listing of current instrumentation and software.

LABORATORY INFORMATION MANAGEMENT SYSTEM

PACE, Inc. uses a state-of-the art Laboratory Information Management System (LIMS). The overall objectives of the LIMS are:

- to improve workload management by providing up-to-date project and sample status information;
- to increase laboratory efficiency through the production of tracking reports, which facilitates "batch" processing across all active projects;
- to give clients direct access to the status of their projects through remote connection to the LIMS, with the use a computer and remote communications software;
- to improve laboratory resource management by producing turnaround time reports for each laboratory;
- to provide consolidated financial information on revenues and operating costs.

The central computer system for the LIMS is an Everex 80386 computer with a 330 megabyte disk drive. The computer uses the UNIX operating system, which supports multitasking operations and simultaneous multiuser access to the LIMS database. Fourteen remote terminals are located throughout the laboratories and offices. The terminals are used to enter new project information, to update active project status and to produce management reports.

Sample shipments are received in the Sample Control Center where they are unpacked and inventoried against the accompanying chain-of-custody documentation. The shipment is then logged into LIMS, which then tracks sample flow through the laboratory from sample receipt, to the generation of the analytical report.

INORGANIC/METALS LABORATORY

The Metals Laboratory is equipped with five major instruments including a Jarrell Ash 9000 (61 upgrade) simultaneous inductively coupled plasma (ICP), a Perkin-Elmer 5100 graphite furnace atomic absorption spectrometer (GFAA), a Perkin-Elmer Z3030 GFAA, a Perkin-Elmer Plasma 40 sequential ICP and a Buck Scientific Mercury Analyzer 400. These instruments comprise the current technology required to analyze EPA regulated metal constituents. Telecations Enviroform, and Smartlog provide the generation of metal report in the EPA data reporting format.

INORGANIC/WATER QUALITY LABORATORY

The Inorganic/Water Quality Laboratory is equipped with a Hach UV-Visible Spectrophotometer used for many analytical colorimeter tests. A Dohrmann DX-20A TOX Analyzer is used to determine the presence of organic halogens. A Dohrmann DC-80 TOC analyzer is used to determine the presence of organic carbon in aqueous and soil samples. A Turner 40 Nephelometer is used to measure turbidity. In addition, the laboratory houses other small equipment such as specific ion electrodes, dissolved oxygen probe, pH meters, adequate glassware, and reagents for classical inorganic water quality tests.

ORGANIC EXTRACTION LABORATORIES

Three separate laboratories are committed to preparing samples for organic analyses. These laboratories contain adequate bench and hood space and are equipped with appropriate glassware for the timely and cost-effective processing of samples prior to analyses. The water laboratory is equipped with more than thirty sets of continuous extractors for the automated processing of liquid samples. The instrumentation laboratory has two Analytical Biochemistry Laboratories Autoprep 1002 A Automated Gel Permeation Chromatograph (GPC) in use, when necessary, for cleanup of samples with complex matrices. Also, a screening HP 5880 GC-FID/ELCD. The soils laboratory is equipped with sonicators. The third laboratory is dedicated to soil preparation. The lab is equipped with 3 soil sonicators, a centrifuge, and other soil preparation supplies. The organic extraction laboratory also analyzes total petroleum hydrocarbons (TPH) with a Perkin Elmer 1320 Infrared Spectrophotometer.

GAS CHROMATOGRAPHY LABORATORY

Two separate Gas Chromatography (GC) Laboratories are dedicated to chromatographic instrumentation. Segregation of the GC volatiles and extractables laboratories minimizes the potential for cross contamination. The laboratories are equipped with a total of eight (8) hp 5890A gas chromatographs which represent the state-of-the-art in environmental instrumentation.

These instruments are microprocessor controlled and are equipped with an hp 7673A and/or Tekmar automatic samplers for efficient sample processing. The GC systems have megabore capillary, split/splitless capillary, and conventional packed column capabilities and are equipped with a wide range of detectors for maximum flexibility in designing analytical approaches to trace environmental analyses.

Each GC laboratory is equipped with a PE Nelson model 2600 chromatography data system supplemented by customized software. Using this PC based system, analog data from the GC detectors are digitized by intelligent interface boxes and uploaded to the PE Nelson model 2600 data system running on a server PC. The data are stored on hard disc and may be processed using Nelson programs to integrate, calibrate and report data. Supplemental programs provide the production of finished reports in the EPA data reporting format and for turnkey quality assurance evaluation. Data access by satellite computers is provided by use of a Local Area Network (LAN) hosted on the Nelson server computer. Long term raw data storage is provided on magnetic tape.

GAS CHROMATOGRAPHY/MASS SPECTROMETRY LABORATORY

Two separate GC/MS laboratories are dedicated to GC/MS instrumentation. The Semi-volatile and VOA Gas Chromatography/Mass Spectrometry (GC/MS) laboratories are equipped with six automated mass spectrometers which represent state-of-the-art instrumentation in environmental service applications. These instruments are equipped with automatic sample introduction equipment to facilitate rapid sample turnaround. Custom computer software for data reduction is used to minimize data handling for many types of analyses.

The GC/MS Department is equipped with a Finnigan 5100 GC/MS/DS system, two Finnigan 4510 systems, two INCOS 50 systems and one INCOS 500 system. Data systems with the equipment include extensive capabilities for automated multi-point calibration and data processing incorporating "Superincos" memory-mapped software. Qualitative identifications are facilitated by using the newest NBS mass spectral library containing more than 45,000 reference spectra for identification of unknown compounds. These GC/MS systems represent the most advanced technology available for repetitive analyses of complex samples. Data samples are interfaced to an IBM PC-XT. The IBM PC-XT is used to produce customized data reports automatically using Finnigan's QA Formaster database manager and report generator.

The GC/MS systems are equipped with both split/splitless and capillary-column chromatography as well as conventional packed-column chromatography capabilities. One Tekmar LCS-2/ALS (automated 10 port sequential sampler) and 3 Tekmar LCS-2000/ALS-2016 purge and trap devices (automated 16 port sequential sampler) are used for the analysis of volatile (purgeable) organics in water and solid matrices. In addition, two Tekmar 2032 automatic sample heaters are available for heated purge and trap analysis. Two A2OOS CTC autosamplers are used for automated Semivolatile analyses. The volatiles and semivolatiles laboratories are segregated to minimize cross-contamination. The volatiles laboratory is equipped with a separate environmental control and air-handling system and separate fume hood to minimize contamination and provide for maximum stability.

Standard Analytical Methods and Procedures

Laboratory Equipment Inventory

Table 4.6

Analytical Equipment	Manufacturer/Model	
Gel Permeation Chromatograph, autoprep (2)	A. B. C. Labs	1002B
GC/MS	Finnigan	5100
GC/MS (2)	Finnigan	4510
GC/MS (2)	Finnigan	Incos 50
GC/MS	Finnigan	Incos 500
GC/MS	Hewlett Packard	5971
GC/MS	Hewlett Packard	5988A
Gas Chromatograph w/Dual ECD (4)	Hewlett Packard	5890A
Gas Chromatograph w/Dual FPD	Hewlett Packard	5890A, 5890SII
Gas Chromatograph w/Dual PID/FID	Hewlett Packard	5890A
Gas Chromatograph w/Dual PID/ELCD (2)	Hewlett Packard	5890A, 5890SII
Gas Chromatograph w/Dual FID (3)	Hewlett Packard	5890A, 5890SII
Gas Chromatograph w/Dual NPD	Perkin Elmer	1320
Gas Chromatograph w/ FID/ECD	Hewlett Packard	5890A
ICP	Perkin-Elmer	P-40
ICP	Jarrell-Ash	9000
Spectrophotometer, Atomic Absorption Graphite Furnace	Perkin-Elmer	3030
Spectrophotometer, Atomic Absorption Graphite Furnace	Perkin-Elmer	5100
Spectrophotometer, Atomic Absorption Graphite Furnace	Perkin-Elmer	4100
Mercury Analyzer (Cold Vapor) (2)	Buck Scientific	400A
Expandable Ion Analyzer	Orion	EA940
Spectrophotometer	Hach	DR 3
Conductance Meter	YSI	031
Nephelometer	Turner	TD-40
TOC Analyzer	Dohrman	DC-80
TOX	Dohrman	MC-3/AD-3
Automated Ion Analyzer	Latchat	
Infrared Spectrophotometer	Perkin Elmer	1320

Standard Analytical Methods and Procedures

Laboratory Equipment Inventory

Table 4.6

Computer and Data Handling Equipment

Incos Data System (7)	Finnigan
Nelson Data System Interface (8)	Perkin-Elmer
Formaster	Finnigan
Enviroforms	Telecations

Miscellaneous Laboratory Support Equipment

Analytical Balances	Water Baths
Top Loading Balances	Hot Plates and stirrers
Deionized Water System	Muffle Furnace
Drying Ovens	Evaporator/Sample Concentrators

SECTION 5

QUALITY ASSURANCE OBJECTIVES

The objectives of the Quality Assurance (QA) efforts for the laboratory activities are twofold. First, they provide the mechanism for ongoing control and evaluation of measurement data quality on a routine basis, (our internal quality assurance programs). Second, quality control data can ultimately be used to define data quality for the various measurement parameters in terms of precision and accuracy. This section describes the QC components and frequencies that are followed.

5.1 QC Components

5.1.1 Trip Blank/Field Blank Analysis

Trip/Field blanks (if specified by the client) shall be analyzed to monitor for possible sample contamination during shipment or field collection. Blanks accompany the sample bottles through collection and shipment to the laboratory and are stored with the samples. Sample Management supplies every volatile organic sample cooler with a trip blank. At the client's request, a trip blank will be sent with any sample cooler. Results of the Trip/Field blanks are often useful in determining the validity or importance of actual sample results. The results of trip blank analyses shall be maintained with the corresponding sample analytical data in the project file.

5.1.2 Method (Laboratory) Blank Analyses

A laboratory blank is a volume of appropriately pure (deionized, distilled) water carried through the entire analytical procedure. The volume of the blank must be approximately equal to the sample volume processed. A method blank shall be performed with each batch of less than or equal to 20 samples processed. Analysis of the blank verifies that method interferences caused by contaminants in solvents, reagents, glassware, and other sample processing hardware are known and minimized. Optimally, a method blank should contain no analytes of interest above the method detection limit for that analyte. There are exceptions, however, for normally observed organic solvents such as methylene chloride and acetone. The specific acceptance criteria for these analytes (or others) are contained within the methods employed, and in the laboratory standard operating procedures. The results of all method blanks are maintained with the analytical data set, and reported with all samples contained in the analytical batch.

5.1.3 Laboratory Duplicate Sample Analysis

Duplicate analyses shall be performed to evaluate the precision of analysis. Results of the duplicate analyses are used to determine the relative percent differences between replicate samples. A duplicate analysis shall be performed at a minimally 5 percent (5%) sample frequency (one duplicate sample for each group of 20 samples). Duplicate analysis results shall be summarized on the quality control data summary forms. The duplicate may be a duplicate matrix spike if required by the analytical method.

5.1.4 Matrix Spike Analyses

To evaluate the effect of the sample matrix upon analytical methodology, sample aliquots shall be spiked with analytes of interest. The concentration of analytes added will be in the range of 5X MDL to 2X sample concentration. The percent recovery for the respective compounds shall then be calculated. If the percent recovery falls outside established quality control limits, the data should be evaluated along with other QC data to determine if the sample should be reanalyzed. Spiked samples shall be run at five percent (5%) sample frequency (one spiked sample for each group of 20 samples). Matrix spike results will be summarized on the quality control data summary forms. Matrix spike recovery criteria for many methods are advisory only and do not require re-analysis.

5.1.5 Surrogate Standard Analyses

Surrogate standard determinations shall be performed on all samples and blanks for GC/MS and GC (except TPH by GC-FID) analyses. Total petroleum hydrocarbon analysis by GC-FID will be analyzed with surrogates if requested. All samples and blanks are fortified with surrogate spiking compounds before purging or extraction to monitor preparation and analysis of samples. Recoveries should meet method specified acceptance criteria, or those which are established as laboratory results. Surrogate standard data will be summarized on the surrogate standard recovery forms and submitted with each data package (as appropriate).

5.1.6 Method Blank Spike Analysis

To evaluate the accuracy of the method as performed, an aliquot of blank water may be spiked with the analytes of interest and taken through the entire sample process. These data help in determining if the method has been performed correctly that day, and if reanalysis is warranted if the matrix spike recoveries are outside the control limits. These analyses are performed on a one per sample batch basis with a majority of the analytical methods. Blank spike recoveries which are within established control limits may be used for validation of batch QC if MS/MSD excursions are observed due to matrix effects. A method blank spike may also be referred to as Laboratory Control Sample or Independent Calibration verification standard in certain sample analyses.

5.1.7 Laboratory Control Sample

An external, independent source material which is subjected to the entire analytical process is used with many of the methods employed to determine the accuracy of the overall process. In regard, it is closely related to the method blank spike. This component is defined within the method reference (i.e. CLP-SOW for Inorganics) or as required for a particular project or program, such as NEESA.

5.2 Data Precision

Precision is usually expressed as Relative Percent Difference (RPD) based on duplicate analyses of a sample. The specific criteria for precision is contain within the methodology SOPs, and are also shown in Tables 8.1 - 8.9. Data precision is addressed in further detail (i.e. calculations) in Section 11 of this QAP.

5.3 Data Accuracy

Accuracy is usually expressed as percent recovery (%R). The specific criteria for accuracy is contained within the methodology SOPs, and are also shown in Tables 8.1 - 8.9 attached. Data accuracy is addressed in further detail (i.e. calculations) in Section 11 of this QAP.

The objectives listed in these tables are based primarily on performance data from method validation studies or EPA established control limits. These are not intended to represent data validation criteria, per se; rather they represent the performance capability of the methods.

5.4 Data Representativeness/Comparability

Data representativeness expresses the degree to which sample data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point or an environmental condition. It is mostly concerned with sampling program design and is demonstrated through the use of field duplicates, field blanks, field rinsates, and trip blanks.

Data comparability will be achieved by following approved, standard analytical procedures, where such exist, and by reporting results in standard units of measure, as suggested the American Chemical Society's publication, "Principles of Environmental Analysis".

5.5 Data Completeness

Completeness is expressed as the percentage of the amount of valid data obtained compared to the amount of data expected. For the percent recovery required to ensure data accuracy, a minimum 90 percent completeness is the goal of the laboratory when samples values are above ten (10) times the MDL and 70 percent when sample values are below ten (10) times the MDL. For metals, however, a minimum 95 percent completeness is the goal of the laboratory when sample values are above 10 times MDL, and 75 percent when values are below 10 times MDL. Conditions which prevent reaching these goals, such as significant sample matrix difficulties, or sample loss, should be reported to the client in a timely fashion to determine whether remedial action should be taken. Precision and accuracy determinations, if outside the QA objectives due to sample-related causes, may be regarded as qualifying or reanalysis at client cost, rather than invalidating, the associated data.

5.6 Method Detection Limits

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. For EPA 600 and 8000 Series organic analysis, PACE, Inc. uses one of the following guidelines for the determinations of MDLs: For methods which have a CLP counterpart, the CLP MDL is reported. For others, the SW-846 MDL is reported, unless a PACE, Inc. MDL study supports a lower MDL.

The laboratory performs annual MDL studies to demonstrate that it can meet or exceed these recommended MDLs and CRQLs. The procedure used by PACE, Inc. for establishing MDLs is described in Appendix B to 40CFR Part 136 "Definition and Procedure for the Determination of the Method Detection Limit - Revision 1.1". This procedure consists of analyzing seven (7) aliquots of a standard spiked at 3 to 5 times the MDL which is taken through all the sample processing steps of the analytical method. The MDL is defined as three times the standard deviation of the mean value for the seven analyses.

As these detection limits are recommended for non-CLP-SOW or SW-846 analyses, the MDL as stated in the published method is utilized.

Method detection limits utilized by PACE, Inc. for metal analyses are those listed in SW-846 methods or CLP-SOW for CLP work. The laboratory performs quarterly instrument detection limit (IDL) studies to demonstrate that it can meet or exceed the recommended MDLs. The procedure used for this determination is found in the EPA Contract Laboratory Program (CLP) Statement of Work for Inorganics.

Method detection limits utilized by PACE, Inc. for water analysis parameters are those listed in the methods found in "EPA Methods for the Chemical Analysis of Water and Wastewaters". Again, these MDLs are taken from the appropriate method recommendations and are verified annually using EPA protocol.

Soil detection limits are matrix dependent. Estimates of MDLs in soils are based on the corresponding MDL in aqueous matrices adjusted for the mass of sample (wet weight), and the volume of digestate or extract solvent used in each analysis.

5.7 Data Quality Objectives

The above data quality objectives and those outlined in the tables in this section, provide guidance for PACE, Inc.'s routine analytical work. Upon client request, other data quality objectives may be specified. These project-specific data quality objectives will be executed by PACE, Inc. through the use of a Special Request (SR) Analysis form which is distributed to all analysts involved.

SECTION 6

SAMPLING PROCEDURES

PACE, Inc. is not a field sample facility and does not partake in any field sampling procedures.

SECTION 7

SAMPLE MANAGEMENT

A stringent chain-of-custody system is vitally important in ensuring the usefulness of measurement data. There must be a documented, traceable link between any given measurement, sample and parameter that it is reported to represent. Without this link, it cannot be proven with any certainty that the measurement in question actually represents a condition that did exist at the indicated time and place. The chain-of-custody system must provide a documented history of each sample. The history must represent a legally acceptable record that covers all aspects of the pre-sampling preparation, sample collection, post-sampling handling, storage, and analysis process. This record should originate with the preparation of any sample containers that are used and should indicate "who did what and when" until final disposal of the sample. The custody procedures used should also ensure that the integrity of the sample is maintained throughout the course of the collection, handling, and analysis process, i.e., that there is no opportunity for inadvertent contamination.

7.1 Sample Custody

Sample custody is an organized scheme for documenting sample history and providing a legally defensible record for the measurement process. This system of documentation is one aspect of the overall internal quality control (QC) system. The types of documentation which are typically associated with environmental measurement programs include:

7.2 Sample Handling

Prior to collecting samples, the collection team will consider the analyses to be performed so that proper sample containers and shipping containers can be assembled and the proper preservatives added to containers. In addition, field logs and record sheets, Chain-of-Custody and Request for Analysis Forms (Table 7.1) will be assembled.

All records required for documentation of field collection will be completed by the field team. Several of the documents that affect laboratory operations are discussed further in this section. The primary document records are the Chain-of-Custody and Request for Analysis.

In addition to initiating the Chain-of-Custody/Request for Analysis form, field personnel will be responsible for uniquely identifying (required on the chain-of-custody form) and labeling samples, providing proper preservation, and packaging samples to preclude breakage during shipment.

7.3 Sample Identification

Using the appropriate documentation, each sample should be labeled to identify:

- Project number/name
- Unique sample number
- Sample location
- Sampling date and time
- Signature of person obtaining the sample
- Method of sample preservation/conditioning
- Analyses requested

Samples will be identified by attaching a tag and a label (Table 7.2) to the container prior to sampling or immediately thereafter. After collection, separation, identification and preservation, the sample will be maintained under Chain-of-Custody procedures discussed below. If a sample is to be split with another laboratory, it will be split in the field immediately after collection to obtain like portions. In situ measurements (flow, pH, temperature, etc.) will be recorded in field log books. The information will include project name and number, location of sample point, date, time, name and signature of person recording the data, observations and remarks. Entries on all sample and field documents will be made in black indelible ink; changes will be crossed out with a single mark and initialed.

pace INCORPORATED

CHAIN OF CUSTODY

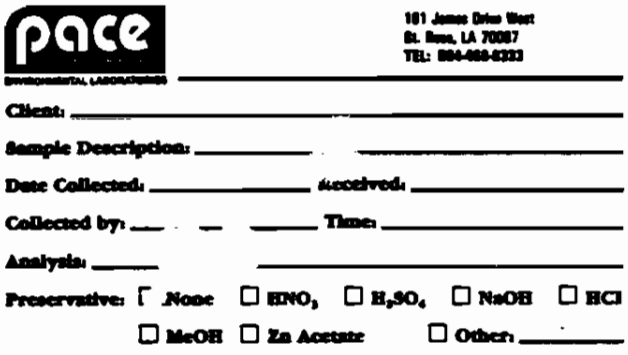
Page _____ of _____

Company Name:	Project Manager:
Project Number:	Project Name:
P. O. #	
Sampled By: (Printed and Written Signature)	

SAMPLE ID	DATE	TIME	MATRIX	LOCATION	TOTAL NO. OF CONTAINERS	ANALYSIS								COMMENTS:

Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	REMARKS:
Relinquished by: (Signature)	Date/Time	Received by: (Signature)	Date/Time	
Relinquished by: (Signature)	Date/Time	Received for Laboratory by: (Signature)	Date/Time	ETC/GULF SOUTH 6801 PRESS DRIVE, EAST BUILDING ORL , LA 6 04)

Figure 7-2. Sample Label



7.4 Sample Controls

Samples will be placed in containers compatible with the intended analysis and properly preserved. Also, collector of samples will consider the time interval between acquiring the sample and analysis (holding time) so that the sample is representative. Table 7.4 provides requirements for various analytical parameters with respect to the type of container, preservation method, and maximum holding time between collection and analysis.

7.5 Chain-of-Custody

The purpose of the chain-of-custody program will be to document sample possession from the time of collection to disposal. The audit trail will consist of not only the Chain-of-Custody document, but also field notebooks, freight bills, internal sample custody forms, analytical benchsheets, analysts notebooks, disposal records and manifests.

7.6 Shipping Procedures

Samples will be accompanied by a Chain-of-Custody Form. When transferring possession of samples, the shipper will sign and note the time on the form. Shipping containers will be closed with strapping tape and custody seals affixed to the front right and back left of each ice chest.

7.7 Laboratory Custody

In the laboratory, a sample custodian is assigned to receive the samples. Upon sample receipt, the custodian will inspect the condition of the custody seals and samples, reconcile the information on the sample labels against that on the Chain-of-Custody, assign laboratory identification numbers, log the samples into the LIMS, and store the samples in a secured sample storage refrigerator. If sample leakage, air bubbles, or documentation discrepancies are found, they will be noted on the Chain-of-Custody, the non-conformance sheet will be noted; and the appropriate Client Service Representative will be notified in order to resolve the discrepancy with the client in a timely manner.

The analyst/technician will carefully record all pertinent sample identification on any benchsheet or analytical notebook used and will use this as documentation of custody. When finished with the sample, the analyst/technician will relinquish possession and place it back in the refrigerator. All sample tracking records, benchsheets, and analytical notebooks will be considered sample custody documentation.

7.8 Sampling

Field sampling is beyond the scope of laboratory operations; however, as stated above, sample containers can be provided by the lab for field sample programs.

All sample containers are purchased from commercial sources and are not reused. The sample containers are supplied and returned from the field in ice chests with their appropriate preservation and packing materials.

15:51:26 PACE/GULF SOUTH 09/16/94

Login Sample Episode: TON

Project: 3572
Starts: 08/24/94
Ends:
Account: [illegible]
Bill Ref:

Contact
Report:
(project)

Mats Key PR 00919

Comments:

Sales 1: 016 -
Terms: Net 30 days
PO#: 5827

Sample No.	Client ID	Analysis	RG# Hold	Dates --> Matrix	COLL	RECV	DUE	Login LI	Subcontr SC	Org Prep OP	GC/MS MS	Gas Chro GC	Metal Pr MP	Metal An MA	Water Qu WQ	Report R RR
		CN-TOT	09/27	Water											WIP	NEED
		COD	10/11	Water											NEED	NEED
		NITRATE	10/11	Water											DONE	NEED
		PHENOLS	10/11	Water											WIP	NEED
		PHOS-HYDRO	10/11	Water											NEED	NEED
		SULFATE	10/11	Water											DONE	NEED
		TDS	09/20	Water											NEED	NEED
		TSS	09/20	Water											NEED	NEED
TON-006	TB.		94152		09/13	09/14	09/26		COMMENTS: 2C							
		8240	09/27	Water							NEED					NEED
TON-007	JE-1		94152		09/13	09/14	09/26		COMMENTS: 10C							
		D/G-IR	10/11	Water						WIP						NEED
		8240	09/27	Water							NEED					NEED
		AS-PP-G	03/12	Water									NEED	NEED		NEED
		MET-PP-I	03/12	Water									NEED	NEED		NEED
		PB-PP-G	03/12	Water									NEED	NEED		NEED
		HG-PP-CV	10/11	Water										NEED		NEED
		AMMONIA-DIST	10/11	Water											NEED	NEED
		CN-TOT	09/27	Water											WIP	NEED
		COD	10/11	Water											NEED	NEED
		NITRATE	10/11	Water											DONE	NEED
		PHENOLS	10/11	Water											WIP	NEED
		PHOS-HYDRO	10/11	Water											NEED	NEED
		SULFATE	10/11	Water											DONE	NEED
		TDS	09/20	Water											NEED	NEED
		TSS	09/20	Water											NEED	NEED
TON-008	JE-2		94152		09/13	09/14	09/26		COMMENTS: 10C							
		D/G-IR	10/11	Water						WIP						NEED
		8240	09/27	Water							NEED					NEED
		AS-PP-G	03/12	Water									NEED	NEED		NEED
		MET-PP-I	03/12	Water									NEED	NEED		NEED
		PB-PP-G	03/12	Water									NEED	NEED		NEED
		HG-PP-CV	10/11	Water										NEED		NEED
		AMMONIA-DIST	10/11	Water											NEED	NEED
		CN-TOT	09/27	Water											WIP	NEED
		COD	10/11	Water											NEED	NEED
		NITRATE	10/11	Water											DONE	NEED
		PHENOLS	10/11	Water											WIP	NEED
		PHOS-HYDRO	10/11	Water											NEED	NEED
		SULFATE	10/11	Water											DONE	NEED
		TDS	09/20	Water											NEED	NEED
		TSS	09/20	Water											NEED	NEED

TABLE 7.4 SAMPLING, PRESERVATION, VOLUME AND HOLDING TIME REQUIREMENTS

PARAMETER	MATRIX	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
Bacterial Tests					
Coliform, fecal and total	W	P,G	200	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	6 Hours
Fecal streptococci	W	P,G	200	Cool 4°C, 0.008% $Na_2S_2O_3$[d]	6 Hours
Inorganic Tests					
Acidity	W	P,G	50	Cool 4°C	14 Days
Alkalinity	W	P,G	50	Cool 4°C	14 Days
Ammonia	W	P,G	100	Cool 4°C, H_2SO_4 to pH<2	28 Days
Biochemical Oxygen Demand	W	P,G	1000	Cool 4°C	48 Hours
Biochemical Oxygen Demand (carbonaceous)	W	P,G	1000	Cool 4°C	48 Hours
Bromide	W	P,G	200	None required	28 Days
Chemical Oxygen Demand	W	P,G	75	Cool 4°C, H_2SO_4 to pH<2	28 Days
Chloride	W	P,G	50	None required	28 Days
Chlorine, Total Residual	W	P,G	200	None required	Analyze Immediately
Color	W	P,G	50	Cool 4°C	48 Hours
Cyanide, Total and Amenable to Chlorination	W	P,G	1500	Cool 4°C, NaOH to pH >12, 0.6 g ascorbic acid[d]	14 Days[e]
Fluoride	W	P	300	None required	28 Days
Hardness	W	P,G	100	HNO_3 to pH <2, H_2SO_4 to pH <2	6 Months
Hydrogen Ion (pH)	W	P,G	25	None required	Analyze Immediately
Kjeldahl and Organic Nitrogen	W	P,G	500	Cool 4°C, H_2SO_4 to pH<2	28 Days

See footnotes at end of table.

TABLE 7.4 SAMPLING, PRESERVATION, VOLUME AND HOLDING TIME REQUIREMENTS

PARAMETER	MATRIX	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
Inorganic Tests					
Chromium VI	W	P,G	50	Cool 4°C	24 Hours
Mercury	W	P,G	100	HNO_3 to pH <2	28 Days
	S	P,G	20 gm	None	28 Days
Metals, Except Chromium VI and Mercury	W[f]	P,G	200	HNO_3 to pH <2	6 Months
	S	P,G	50 gm	None	6 Months
Nitrate	W	P,G	100	Cool 4°C	48 Hours
Nitrate-Nitrite	W	P,G	100	Cool 4°C, H_2SO_4 to pH<2	28 Days
Nitrite	W	P,G	50	Cool 4°C	48 Hours
Oil and Grease	W	G	1000	Cool 4°C, H_2SO_4 to pH<2	28 Days
Organic Carbon	W	P,G	25	Cool 4°C, HCl or H_2SO_4 to pH<2	28 Days
Orthophosphate	W	P,G	50	Filter immediately, Cool 4°C	48 Hours
Oxygen Dissolved Probe	W	G Bottle and top	300	None required	Analyze immediately
Phenols	W	G	500	Cool 4°C, H_2SO_4 to pH<2	28 Days
Phosphorus (Elemental)	W	G	50	Cool 4°C	48 Hours
Phosphorus, Total	W	P,G	50	Cool 4°C, H_2SO_4 to pH<2	28 Days
Residue, Total	W	P,G	100	Cool 4°C	7 Days
Residue, Filterable	W	P,G	100	Cool 4°C	48 Hours
Residue, Nonfilterable	W	P,G	250	Cool 4°C	7 Days
Residue, Settleable	W	P,G	1000	Cool 4°C	48 Hours
Residue, Volatile	W	P,G	100	Cool 4°C	7 Days
Silica	W	P	50	Cool 4°C	28 Days
Specific Conductance	W	P,G	100	Cool 4°C	28 Days

See footnotes at and of table.

TABLE 7.4 SAMPLING, PRESERVATION, VOLUME AND HOLDING TIME REQUIREMENTS

PARAMETER	MATRIX	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
Inorganic Tests (continued)					
Sulfate	W	P,G	100	Cool 4°C	28 Days
Sulfide	W	P,G	500	Cool 4°C, add zinc acetate + sodium hydroxide to pH >9	7 Days
Sulfite	W	P,G	50	None required	Analyze immediately
Surfactants	W	P,G	250	Cool 4°C	48 Hours
Temperature	W	P,G	1000	None required	Analyze immediately
Turbidity	W	P,G	100	Cool 4°C	48 Hours
Organic Tests[g]					
Purgeable Halocarbons	W	G,Teflon-lined septum	40	Cool 4°C, 0.008% $Na_2S_2O_3$[e]	14 Days
	S	G,Teflon-lined septum	40 gms	Cool 4°C	14 Days
Purgeable Aromatic	W	G,Teflon-lined septum	40	Cool 4°C, 0.008% $Na_2S_2O_3$[e] HCl to pH 2[h]	14 Days
Hydrocarbons	S	G,Teflon-lined septum	40 gms	Cool 4°C	14 Days
Volatile Organics	W	G,Teflon-lined septum	40	Cool 4°C, HCl to pH 2[h]	14 Days
	S	G,Teflon-lined septum	40 gms	Cool 4°C	14 Days
Volatile Organics by EPA CLP	W	G,Teflon-lined septum	40	Cool 4°C	10 Days from VTSR[ik]
	S	G,Teflon-lined septum	40 gms	Cool 4°C	10 Days from VTSR[ik]
Phenols[j]	W	G,Teflon-lined cap	1000	Cool 4°C, 0.008% $Na_2S_2O_3$[e]	7 Days until extraction
	S	G,Teflon-lined cap	100 g	Cool 4°C	40 Days after extraction
Pesticides/PCBs[j]	W	G,Teflon-lined cap	1000	Cool 4°C	7 Days until extraction
	S	G,Teflon-lined cap	100 g	Cool 4°C	40 Days after extraction
Pesticides/PCBs by EPA CLP	W	G,Teflon-lined cap	1000	Cool 4°C	5 Days from VTSR
	S	G,Teflon-lined cap	100 g	Cool 4°C	35 Days after extraction

See footnotes at end of table.

TABLE 7.4 SAMPLING, PRESERVATION, VOLUME AND HOLDING TIME REQUIREMENTS

PARAMETER	MATRIX	CONTAINER[a]	VOLUME REQUIRED (mL)	PRESERVATION[b]	MAXIMUM HOLDING TIMES[c]
Organic Tests[d] (Continued)					
Semivolatiles	W	G,Teflon-lined cap	1000	Cool 4°C	7 Days until extraction
					40 Days until analysis
	S	G,Teflon-lined cap	100 g	Cool 4°C	14 Days until extraction
					40 Days until analysis
Semivolatiles by EPA CLP	W	G,Teflon-lined cap	1000	Cool 4°C	5 Days from VTSR
					35 Days until analysis
	S	G,Teflon-lined cap	100 g	Cool 4°C	10 Days from VTSR
					35 Days until analysis

REFERENCE: This table includes the requirements of the U.S. Environmental Protection Agency, as published in the Code of Federal Regulations, Vol. 49, No. 209, 40 CFR 136, October 26, 1984, pg. 43260, EPA SW846, 3rd Edition, and EPA CLP SOWs.

[a] Polyethylene (P) or glass (G).

[b] Sample preservation should be performed immediately upon sample collection. For composite chemical samples, each aliquot should be preserved at the time of collection. When use of an automatic sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

[c] Samples should be analyzed as soon as possible after collection. The times listed are maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if permittee, or monitoring laboratory, has data on file to show that the specific types of samples under study are stable for the longer time. Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter period if knowledge exists to show this is necessary to maintain sample stability. All dates are from collection unless otherwise specified.

TABLE 7.4 SAMPLING, PRESERVATION, VOLUME AND HOLDING TIME REQUIREMENTS
(Continued)

[d] Should only be used in the presence of residual chlorine.

[e] Maximum holding time is 24 hours when sulfide is present. Optionally, all samples may be tested with lead acetate paper before pH adjustment to determine if sulfide is present.

[f] If dissolved metals are required, filtration prior to acidifying is required.

[g] Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[h] Sample receiving no pH adjustment must be analyzed within seven days of sampling.

[i] EPA CLP samples are usually preserved which extends their holding time.

[j] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6-9. Samples preserved in this manner may be held for seven days before extraction and 40 days after extraction. Exceptions to the optional preservation and holding time procedure are noted in footnote [d] (Ref. the requirement for thiosulfate reduction of residual chlorine) and footnotes [k] and [l] (Ref. the analysis of benzidine.)

[k] For EPA CLP contract protocols, holding times are from VTSR - Verified Time of Sample Receipt.

SECTION 8

ANALYTICAL PROCEDURES AND CALIBRATION

Analytical procedures and calibration are described in this section. Tables are provided to state the QC recovery limits, the Relative Percent Difference (RPD) allowed between duplicate runs, and Method Detection Limit (MDL) for our routine analytical procedures. For more detailed information on method QC requirements, please contact your PACE, Inc. client service representative.

8.1 Laboratory Standards and Reagents

For organic analysis, analytical standards are obtained from two different commercial sources. These sources are checked against each other to ensure quality analytical standards. Both spectral and reagent grade solvents are confirmed, on a lot basis, free from target analyte contamination before use. Standards and reagents are continuously checked through the use of preparation blanks and preparation blank spike standards.

For inorganic analysis, standard reference materials are obtained from two different commerical sources. These standards are checked against each other to verify the reliability of the analytical standards. The process for second source verification specified in the CLP SOW is used. Analytical grade acid and reagents are confirmed, on a lot basis, free from target analyte contamination before use. Standards and reagents are continuously check through the use of method preparation blanks and blank spikes.

All standards and laboratory reagents are dated upon receipt. Source standards are recorded in a bound laboratory notebook. The preparation and use of all standards are recorded in bound laboratory notebooks which document standard traceability to EPA or NBS standards. Additional information recorded includes data of preparation, concentration and name of the preparer and date of expiration.

8.2 Sample Preparation Methods

8.21 Organic Sample Preparation Methods

SW3510 - Continuous Liquid- Liquid Extraction is designed to quantitatively extract nonvolatile and semivolatile organic compounds from liquid samples using separatory funnel techniques. The sample and extracting solvent must be immiscible in order to yield recovery of target compounds. Subsequent cleanup and detection methods are described in the organic analytical method that will be used to analyze the extract. Samples are adjusted to a specified extraction pH and extracted with the appropriate solvent for the analytical method. Methylene chloride is used as the solvent when another solvent is not specified. Samples are extracted three times, and the combined extracts are dried with anhydrous sodium sulfate and concentrated in a Kuderna-Danish apparatus.

SW3520 - Continuous Liquid- Liquid Extraction is designed to quantitatively extract nonvolatile and semivolatile organic compounds from liquid samples using continuous liquid-liquid extractors. The sample and extracting solvent must be immiscible in order to yield recovery of target compounds. Subsequent cleanup and detection methods are described in the organic analytical method that will be used to analyze the extract. Samples are adjusted to a specified extraction pH and extracted with the appropriate solvent for the analytical method. Methylene chloride is used as the solvent when another solvent is not specified. Samples are extracted for 18 hours and the extracts are dried with anhydrous sodium sulfate and concentrated in a Kuderna-Danish apparatus.

SW3540 - Soxhlet Extraction is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. The Soxhlet extraction process ensures intimate contact of the sample matrix with the extraction solvent. Extraction is accomplished by mixing the solid sample with anhydrous sodium sulfate, placing it in an extraction thimble or between two plugs of glass wool, and extracting it with an appropriate solvent in the Soxhlet extractor. Methylene chloride is used as the solvent when another solvent is not specified. The extract is dried and concentrated, and then treated using a cleanup method, or analyzed directly by the appropriate measurement technique.

SW3550 - Sonication Extraction is a procedure for extracting nonvolatile and semivolatile organic compounds from solids such as soils and sludges. The sonication process ensures intimate contact of the sample matrix with the extraction solvent.

A weighed sample of the solid waste is mixed with the extraction media, then dispersed into the solvent using sonication. The extract is then dried with anhydrous sodium sulfate and concentrated with a Kuderna-Danish apparatus. The resulting solution may then be cleaned up or analyzed directly using the appropriate technique. Methylene chloride is typically used as the solvent, although other solvents may be used for specific analytical applications.

SW3580 - Waste Dilution is a solvent dilution of a non-aqueous waste sample prior to cleanup and/or analysis. It is designed for wastes that may contain organic chemicals at a level greater than 20,000 ug/Kg, and that are soluble in the dilution solvent.

SW5030 - Purge-and-Trap Sample Introduction Method is used to determine the concentration of volatile organic compounds in a variety of liquid and solid waste matrices. It is based upon a purge and trap gas chromatographic procedure. The method is applicable to nearly all types of samples, including aqueous sludges, caustic liquors, acid liquors, waste solvents, oily wastes, water, mousses, tars, fibrous wastes, polymeric emulsions, filter cases, spent carbons, spent catalysts, soils and sediments. The success of this method depends on the level of interferences in the sample; results may vary due to the large variability and complexity of matrices of solid waste samples.

A portion of the solid sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanol solution is combined with water in a purging chamber. An inert gas is then bubbled through the solution at ambient temperature to transfer the volatile components to the vapor phase. The vapor is swept through a sorbent column where the volatile components are trapped. After purging is completed, the sorbent column is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. For Methods 8020 and 8030, drying of the trap for four minutes under helium flow is required. The gas chromatographic column is heated to elute the components which are detected by the appropriate detector (Methods 8010, 8020, 8030, 8240).

8.22 Inorganic Sample Preparation Methods

SW3050 - Acid Digestion for Solids, Sediments, and Sludges for Metals Determinations is applicable to the preparation of sediment, sludge, and soil samples for metals analysis by flame (FLAA) or furnace (GFAA) atomic absorption spectrophotometry or by inductively coupled argon plasma emission spectroscopy (ICPES). Laboratories are to digest solid samples for metals analysis by either Method 3050 or 3050R, dependent upon which analytes are of interest.

A 1 g (wet weight) sample is treated and digested in nitric acid and hydrogen peroxide. The digestate is then refluxed with nitric or hydrochloric acid, depending on the type of analysis to be performed. When using HCl as the final refluxing acid, do not boil because antimony is volatile and easily lost. A separate sample is dried for a total solids and/or percent moisture determination.

Some sludge samples can contain diverse matrix types, which may present specific analytical problems. Spiked samples and any relevant standard reference material should be processed as an aid in determining whether Method 3050 is applicable to a given waste.

SW1311 - Toxicity Characteristic Leaching Procedure is employed to determine whether a waste exhibits the toxicity leaching characteristics. The procedure includes a leading extraction for semivolatiles and metals and a zero-headspace extraction for volatile components.

SW3020 - Acid Digestion for Metals prepares waste samples for total metals determination by graphite furnace AA spectroscopy. The samples are vigorously digested with nitric acid followed by dilution with nitric acid.

This method is an acid digestion procedure used for preparation of waste samples for metals analysis. The digested samples can be analyzed for total recoverable and dissolved metals determination by graphite furnace (GFAA) atomic absorption spectrophotometry.

For analysis of total recoverable metals, the entire sample is acidified at collection time with nitric (HNO_3) acid. At the time of analysis, the sample is heated with acid and reduced to a specific volume. The sample must not be boiled because antimony is volatile and easily lost. The digestate is then filtered and diluted to the desired concentration for analysis.

For analysis of dissolved metals, the samples are filtered through a 0.5 um filter immediately upon collection, and acidified with nitric (HNO_3) acid. For analysis, the sample is heated with acid and reduced in volume. The digestate is again filtered (if necessary) and diluted to volume.

SW3010 - Acid Digestion for Metals prepares waste samples for total metal determination by flame AA and ICPES. The samples are vigorously digested with nitric acid followed by dilution with hydrochloric acid.

This method is an acid digestion procedure used for preparation of waste samples for metals analysis. The digested samples can be analyzed for total recoverable and dissolved metals determination by either flame atomic absorption spectrophotometry (FLAA) or inductively coupled argon plasma emission spectroscopy (ICPES).

For analysis of total recoverable metals, the entire sample is acidified at collection time with nitric (HNO_3) acid. At the time of analysis, the sample is heated with acid and reduced to a specific volume. The sample must not be boiled because antimony is volatile and easily lost. The digestate is then filtered and diluted to the desired concentration for analysis.

For analysis of dissolved metals, the samples are filtered through a 0.5 um filter immediately upon collection, and acidified with nitric (HNO_3) acid. For analysis, the sample is heated with acid and reduced in volume. The digestate is again filtered (if necessary) and diluted to volume.

8.3 Organic Analytical Procedures and Calibration

8.31 Gas Chromatography Analytical Methods

601/SW8010 - Halogenated Volatile Organics use the purge and trap method 5030. An inert gas is bubbled through a water matrix to transfer the volatile halocarbons from the liquid to the vapor phase. Halocarbons are removed from the inert gas by passing it through a sorbent trap which is then backflushed into a gas chromatographic column with an electrolytic conductivity detector to separate and quantify the compounds of interest. Medium level soil samples are analyzed by extraction of 4 gm of the sample with 10 mls of methanol and diluting to 5-10 ml of reagent water. Low level soil samples may be analyzed by weighing 1 gm of sample directly into the purge and trap device.

The methods provide for the use of a second gas chromatographic column of dissimilar phase to resolve compounds of interest from interferences that may occur. All instruments are configured for simultaneous second column confirmation for this method. All hits reported must be present on both columns within the specified retention time window in order to be reported.

Calibration - Calibration standards are prepared at five concentration levels in reagent water by dilution of stock standards. Optionally, only three standards may be used for reporting under method 601. The average calibration factor is acceptable as a quantitation column if the RSD does not exceed 20% (maximum 30% for selected analytes), otherwise the column may be used only for confirmation purposes. The initial calibration mean retention time is used to define the target retention time window for the identification of each analyte. The initial calibration mean response factor is used to quantitate subsequent samples.

A continuing calibration is performed every 10 sample runs at the concentration equal to the mid-point of the initial calibration. The continuing calibration is acceptable for a quanititation column if the retention time of an analyte falls within the window established by the initial calibrationm and the difference of the continuing calibration response factor from the inital the initial calibration mean response factor is not more than 15%. The continuing valibration is acceptable for a confirmation column if the retention time of an analyte falls within the window estabished by the initial calibration, and the difference of the continuing response factor from the initial calibration mean response factor is not more than 20%.

CADHS-Total Petroleum Hydrocarbons - Gasoline, diesel, jet fuel, kerosene, motor oil and other similar petroleum hydrocarbons are analyzed by a modified 8015 Samples are extracted via standard EPA method 3510 or 3550 and analyzed on a gas chromatograph equipped with a capillary or megabore column and a FID detector, although this gives qualitative data only.

602/SW8020 - Aromatic Volatile Organics in water and soil samples are analyzed using Methods 602 and 8020. These methods, are also known as BTEX, since the compounds of interest include benzene, toluene, and xylene. These are purge and trap gas chromatographic methods. An inert gas is bubbled through a water matrix to transfer the volatile aromatic hydrocarbons from the liquid to the vapor phase. The aromatics are removed from the inert gas by passing it through a sorbent trap which is then backflushed into a gas chromatographic column with a photoionization detector to separate and quantify the compounds of interest. Medium level soil samples are analyzed by extraction of 4 gm of the sample with 10 ml of methanol and diluting to 5-10 ml of reagent water. Low level soil samples may be analyzed by weighing 1 gm of sample directly into the purge and trap device.

The methods provide for a second chromatographic column of dissimilar phase to resolve compounds of interest from interferences that may occur. All instruments are configured for simultaneous second column confirmation for this method. All hits reported must be present on both columns within the specified retention time window in order to be reported.

Calibration - Calibration standards are prepared at five (for 8010) concentration levels in reagent water by dilution of stock standards. Optionally, only three standards may be used for reporting under method 602. The average calibration factor is acceptable as a quantitation column if the RSD does not exceed 20% (maximum 30% for selected analytes), otherwise the column may be used only for confirmation purposes. The initial calibration mean retention time is used to define the target retention time window for the identification of each analyte. The initial calibration mean response factor is used to quantitate subsequent samples.

A continuing calibration is performed every 10 sample runs at the concentration equal to the mid-point of the initial calibration. The continuing calibration is acceptable for a quanititation column if the retention time of an analyte falls within the window established by the initial calibrationm and the

difference of the continuing calibration response factor from the inital the initial calibration mean response factor is not more than 15%. The continuing valibration is acceptable for a confirmation column if the retention time of an analyte falls within the window estabished by the initial calibrat. n, and the difference of the continuing response factor from the initial calibration mean response factor is not more than 20%.

604/SW8040 - Phenols involve acidification of the sample and extraction of the phenols with methylene chloride. The phenols are separated and quantified by gas chromatography with flame ionization detection.

The method provides for a second gas chromatographic column of dissimilar phase to resolve compounds of interest from interferences that may occur. All instruments are configured for simultaneous second column confirmation for this method. All hits reported must be present on both columns within the specified retention time window in order to be reported.

Calibration - Calibration standards are prepared at five concentration levels in a reagent by dilution of stock standards. Optionally, only three standards may be used for reporting under method 604. The average calibration factor is acceptable as a quantitation column if the RSD does not exceed 20% (maximum 30% for selected analytes), otherwise the column may be used only for confirmation purposes. The initial calibration mean retention time is used to define the target retention time window for the identification of each analyte. The initial calibration mean response factor is used to quantitate subsequent samples.

A continuing calibration is performed every 10 sample runs at the concentration equal to the mid-point of the initial calibration. The continuing calibration is acceptable for a quanititation column if the retention time of an analyte falls within the window established by the initial calibrationm and the difference of the continuing calibration response factor from the inital the initial calibration mean response factor is not more than 15%. The continuing valibration is acceptable for a confirmation column if the retention time of an analyte falls within the window estabished by the initial calibration, and the difference of the continuing response factor from the initial calibration mean response factor is not more than 20%.

SW8140 - use the purge and trap method 5030. An inert gas is bubbled through a water matrix to transfer the volatile halocarbons from the liquid to the vapor phase. Halocarbons are removed from the inert gas by passing it through a sorbent trap which is then backflushed into a gas

chromatographic column with an electrolytic conductivity detector to separate and quantify the compounds of interest. Medium level soil samples are analyzed by extraction of 4 gm of the sample with 10 mls of methanol and diluting to 5-10 ml of reagent water. Low level soil samples may be analyzed by weighing 1 gm of sample directly into the purge and trap device.

The methods provide for the use of a second gas chromatographic column of dissimilar phase to resolve compounds of interest from interferences that may occur. All instruments are configured for simultaneous second column confirmation for this method. All hits reported must be present on both columns within the specified retention time window in order to be reported.

Calibration - Calibration standards are prepared at five concentration levels in reagent water by dilution of stock standards. Optionally, only three standards may be used for reporting under method SW8140. The average calibration factor is acceptable as a quantitation column if the RSD does not exceed 20% (maximum 30% for selected analytes), otherwise the column may be used only for confirmation purposes. The initial calibration mean retention time is used to define the target retention time window for the identification of each analyte. The initial calibration mean response factor is used to quantitate subsequent samples.

A continuing calibration is performed every 10 sample runs at the concentration equal to the mid-point of the initial calibration. The continuing calibration is acceptable for a quanititation column if the retention time of an analyte falls within the window established by the initial calibrationm and the difference of the continuing calibration response factor from the inital the initial calibration mean response factor is not more than 15%. The continuing valibration is acceptable for a confirmation column if the retention time of an analyte falls within the window estabished by the initial calibration, and the difference of the continuing response factor from the initial calibration mean response factor is not more than 20%.

615/SW8150 - Chlorinated Herbicides is a gas chromatographic (GC) method for determining certain chlorinated acid herbicides. Spiked samples are used to verify the applicability of the chosen extraction technique to each new sample type. The esters are hydrolyzed with potassium hydroxide, and extraneous organic material is removed by a solvent wash. After acidification, the acids are extracted with solvent and converted to their methyl esters using diazomethane as the derivatizing agent. After any excess reagent is removed, the esters are identified by gas chromatography employing an electron capture detector. The results are reported as the acid equivalents.

All instruments are configured for simultaneous second column confirmation for this method. All hits reported must be present on both columns within the specified retention time window in order to be reported.

Calibration - External standard quantitation method will be used to quantitate all herbicides. Calibration standards are prepared at five concentration levels in a reagent by dilution of stock standards. Optionally, only three standards may be used for reporting under method 615. The average calibration factor is acceptable as a quantitation column if the RSD does not exceed 20% (maximum 30% for selected analytes), otherwise the column may be used only for confirmation purposes. The initial calibration mean retention time is used to define the target retention time window for the identification of each analyte. The initial calibration mean response factor is used to quantitate subsequent samples.

A continuing calibration is performed every 10 sample runs at the concentration equal to the mid-point of the initial calibration. The continuing calibration is acceptable for a quanititation column if the retention time of an analyte falls within the window established by the initial calibrationm and the difference of the continuing calibration response factor from the inital the initial calibration mean response factor is not more than 15%. The continuing valibration is acceptable for a confirmation column if the retention time of an analyte falls within the window estabished by the initial calibration, and the difference of the continuing response factor from the initial calibration mean response factor is not more than 20%.

608/SW8080/EPA SOW - Organochlorine Pesticides and PCBs are analyzed by following SW-846, Method 8080, 3rd Ed. with EPA CLP modifications or by strict EPA CLP, depending on client requirements. Method 8080 is used to determine the concentration of various organochlorine pesticides and polychlorinated biphenyls (PCBs). Prior to the use of this method, samples will be extracted following procedures outlined in SW-846, 3rd Ed. Both neat and diluted liquids may be analyzed by direct injection.

The method provides for a second gas chromatographic column of dissimilar phase to resolve compounds of interest from interferences that may occur. All instruments are configured for simultaneous second column confirmation for this method. All hits reported must be present on both columns within the specified retention time window and be withing a factor of two in order to be reported. If analyte concentration exceeds a factor of two difference then interferences are suspect and flagged or narrated as such. Breakdown of 4,4'-DDT and endrin will also be monitored. Breakdown may not exceed 20%.

Calibration for 608/SW8080 - The external standard quantitation discussed in the method is used to quantitate all pesticides/PCBs. Calibration standards are prepared at five concentration levels in reagent hexane by dilution of stock standards. Optionally, only three standards may be used for reporting under method 608. The average calibration factor is acceptable as a quantitation column if the RSD does not exceed 20% (maximum 30% for selected analytes), otherwise the column may be used only for confirmation purposes. The initial calibration mean retention time is used to define the target retention time window for the identification of each analyte. The initial calibration mean response factor is used to quantitate subsequent samples.

A continuing calibration is performed every 10 sample runs at the concentration equal to the mid-point of the initial calibration. The continuing calibration is acceptable for a quanititation column if the retention time of an analyte falls within the window established by the initial calibrationm and the difference of the continuing calibration response factor from the inital the initial calibration mean response factor is not more than 15%. The continuing valibration is acceptable for a confirmation column if the retention time of an analyte falls within the window estabished by the initial calibration, and the difference of the continuing response factor from the initial calibration mean response factor is not more than 20%.

Calibration for EPA-CLP - Calibration and analysis is performed strictly according to the requested Organic SOW.

8.32 GC/MS Analytical Methods

624 (CLP Mod)/SW8240/EPA SOW - Volatile Organics are analyzed by scanning gas chromatography/mass spectrometry (GC/MS) following SW846 Method 8240, 3rd Edition with EPA CLP modifications or by strict EPA CLP depending on client requirements. Analyte identification and quantitation are performed using response factors and retention times generated from a five-point calibration curve, relative to the closest eluting of three internal standards. The three internal standards are:

- Bromochloromethane
- 1,4-Difluorobenzene
- Chlorobenzene-d_5

Upon client request, tentatively identified compounds may be analyzed and subsequently identified only when a good match is obtained between the unknown spectra and the library spectra. The tentatively identified compound is then quantitated using a response factor of 1.0, with respect to the closest eluting internal standard.

Calibration - The mass spectrometer is tuned daily and every 12 hours to give an acceptable spectrum for bromofluorobenzene (BFB). Relative ion abundance criteria for BFB are given in SW846.

System performance is verified initially and after every 12 hours to ensure a minimum average response factor of 0.3 (0.25 for bromoform) for the following system performance check compounds (SPCCs):

- Chloromethane
- 1,1-Dichloroethane
- Bromoform
- 1,1,2,2-Tetrachloroethane
- Chlorobenzene

A 5-point calibration, used for generating response factors, is performed initially using 10, 25, 40, 60, and 80 ug/L standards. The relative standard deviation (RSD) must be less than 30 percent for the five response factors calculated for each of the following calibration check compounds (CCCs):

- 1,1-Dichloroethene
- Chloroform
- 1,2-Dichloropropane
- Toluene
- Ethylbenzene
- Vinyl chloride

The continuing (every 12 hours) calibration check is performed, following the system performance check, using the CCCs listed above. A single concentration of each CCC is analyzed and a response factor calculated. The single-point RF for each CCC must be within 25 percent of the average five-point RF; otherwise, a new five-point calibration must be generated.

625 (CLP Mod)/SW8270/EPA SOW semivolatile extracts are analyzed by gas chromatography/mass spectrometry following SW846 Method 8270, 3rd Edition with EPA CLP modifications. All samples are prepared following extraction procedures outlined in either 625 or SW846. Identification and quantitation are performed using response factors and retention times generated from a five-point calibration curve, relative to the closest eluting of six internal standards. The six internal standards are:

- Dichlorobenzene-d_4
- Naphthalene-d_8
- Acenaphthene-d_{10}
- Phenanthrene-d_{10}
- Chrysene-d_{12}
- Perylene-d_{12}

Upon client request tentatively identified compounds may be analyzed and are subsequently identified only when a good match is obtained between the unknown spectra and the library spectra. The tentatively identified compound is then quantitated using a response factor of 1.0, with respect to the closest eluting internal standard.

Calibration - The mass spectrometer is tuned daily and every 12 hours to give an acceptable spectrum for DFTPP. DFTPP ion abundance criteria are given in SW846.

System performance is verified initially and after every 12 hours to ensure a minimum average response factor of 0.050 for the following system performance check compounds (SPCCs):

- N-nitroso-di-n-propylamine
- Hexachlorocyclopentadiene
- 2,4-Dichlorophenol
- 4-Nitrophenol

A five-point calibration, used for generating response factors, is performed initially using 20, 50, 80, 120, and 160 ug/L standards. The variability for specific ion response factors for Method 8270 calibration check compounds must be less than 30 percent RSD over the range calibrated. The CCCs are:

- Phenol
- 1,4-Dichlorobenzene
- 2-Nitrophenol
- 2,4-Dichlorophenol
- Hexachlorobutadiene
- 4-Chloro-3-methylphenol
- Acenaphthene
- 2,4,6-Trichlorophenol
- N-nitroso-di-n-phenylamine
- Pentachlorophenol
- Fluoranthene
- Di-n-octylphthalate
- Benzo(a)pyrene

A continuing (every 12 hours) calibration check is performed, following the system performance check, using the CCCs listed above. A single concentration of each CCC is analyzed and a response factor calculated. The single-point RF for each CCC must be within 30 percent of the average five-point RF; otherwise, a new five-point calibration must be generated.

8.4 Inorganic Analytical Procedures and Calibration

8.41 Trace Metals Analytical Methods

SW6010/CLP-SOW - Metals by ICPES describe the simultaneous, or sequential, determination of elements using inductively coupled plasma atomic emission spectroscopy. The method measures element-emitted light by optical spectrometry. Samples are nebulized, and the resulting aerosol is transported to the plasma torch. Element specific atomic-line emission spectra are produced which are dispersed by a grating spectrometer and monitored for intensity by photomultiplier tubes.

Calibration - Detailed calibration procedures for ICPES systems are described in SW846, 3rd Edition and the EPA SOW. A response factor is calculated daily for each metal based on three exposures of a calibration standard and calibration blank. The RF is calculated and stored in the ICPES computer. Following calibration, a mid-level calibration check sample is analyzed; agreement between the measured value and the expected value must be within 10 percent for analyses to proceed. Calibration is verified by analyzing a QC check standard (prepared independently of calibration standards) every 10 samples; agreement within $\pm$10 percent of the expected value is required for all metals analyzed by ICPES.

Metals by AAS - SW7060, 7421, 7740, 7841, 7470, 7471 and the CLP-SOW the above methods are graphite furnace atomic absorption (GFAA) techniques for determination of arsenic, lead, selenium, and thallium, respectively. Following sample digestion, an aliquot of sample is placed in a graphite tube in the furnace, evaporated to dryness, charred, and atomized. The metal atoms to be measured are placed in the light path of an atomic absorption spectrophotometer.

Method 7470-7471 is a cold-vapor atomic absorption procedure for determining the concentration of mercury in waste samples. Sample preparation is specified in the method. Following dissolution, mercury in the sample is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer.

Calibration - The calibration procedures for the graphite furnace and cold vapor AAS systems are described in the respective method in SW846, 3rd Edition and CLP-SOW. A multipoint calibration curve is generated for each element using a calibration blank and three upscale standards. The correlation coefficient for the linear regression equation must exceed 0.995 to be acceptable. Calibration will be verified every 10 samples by analyzing a QC check sample and calibration blank. Agreement within $\pm$10 percent of the expected value is required; otherwise a new calibration curve must be generated.

8.42 Water Quality Analytical Methods

130.2 - Hardness is a measure of the capacity of water to precipitate soap, caused chiefly by the presence of calcium and magnesium ions. Total hardness is defined as the sum of the calcium and magnesium ions, expressed as calcium carbonate.

In this method, the calcium and magnesium ions become complexed upon the addition of ethylenediamine tetraacetate (EDTA) by titration. The solution changes from red to blue when the ions are completely complexed.

Calibration - The EDTA titrant is standardized daily against reagent calcium carbonate. A quality control check sample will be analyzed once per batch.

150.1 - pH Method is used to measure the pH of aqueous wastes and those multiphase wastes where the aqueous phase constitutes as least 20% of the total volume of the waste. The pH of the sample is determined electrometrically using either a glass electrode in combination with a reference potential or a combination electrode.

Calibration - The measuring device is calibrated using a minimum of two standard buffer solutions. The reading must be within 0.05 pH units of each of the buffer solution values.

160.2 - Residue, Filterable (TSS) is applicable to drinking, surface and saline waters, domestic and industrial wastes. A well-mixed sample is filtered through a standard glass fiber filter. The residue retained on the tared filter is dried and determined gravimetrically.

Calibration - The analytical balance must be checked daily with standard weights. The balance readings must agree within 0.001 grams.

180.1 - Turbidity is based upon a comparison of the intensity of light scattered by the sample under defined conditions with the intensity of light scattered by a standard reference suspension. The higher the intensity of scattered light, the higher the turbidity. Readings, in NTUs are made in a nephelometer designed according to specifications outlined in the method.

Calibration - A standard suspension of Formazin, prepared under closely defined conditions, is used to calibrate the instrument. A quality control check sample will be analyzed for every 20 samples; recovery must be within $\pm$15 percent of the expected value.

310.1 - Alkalinity An unaltered sample is titrated to an electrometrically determined end point of pH 4.5. The sample must not be filtered, diluted, concentrated or altered in any way.

Calibration - The acid titrant is standardized daily against reagent sodium carbonate. A quality control check sample will be analyzed once per 20 samples; recovery must be within ±15 percent of the expected value.

325.3 - Chloride provides instructions for the titrimetric determination of chloride using mercuric nitrate and diphenylcarbazonebromophenol indicators.

Calibration - The mercuric nitrate titrant is standardized daily before use against sodium chloride. The titrant is checked against quality control samples and deemed acceptable if the recovery is within 10 percent of the expected value.

335.2/SW9010/EPA SOW - Total and Amenable Cyanide are used to determine the concentration of inorganic cyanide in an aqueous waste or leachate. The methods detect inorganic cyanides that are present as either sample soluble salts or complex radicals. They are used to determine values for both total cyanide and cyanide amenable to chlorination. The cyanide, as hydrocyanic acid (HCN), is released by refluxing the sample with strong acid and distillation of the HCN into an absorber-scrubber containing sodium hydroxide solution. The cyanide ion in the absorbing solution is then determined by automated UV colorimetry.

Calibration - A daily calibration curve must contain a minimum of a blank and three standards, with a correlation coefficient greater than 0.995. A quality control check sample should be analyzed every 20 samples.

350.2 - Ammonia covers the determination of ammonia in drinking, surface, and saline waters, domestic and industrial wastes in the range of 0.01 to 2.0 mg/L NH_3 as N. This range is for photometric measurements made at 630-660 nm in a 15 mm or 50 mm tubular flow cell. Higher concentrations can be determined by sample dilution. Alkaline phenol and hypochlorite react with ammonia to form indophenol blue that is proportional to the ammonia concentration. The blue color is intensified with sodium nitroprusside.

Calibration - A calibration curve will be generated using a reagent blank and three or more standards on a daily basis. The correlation coefficient must exceed 0.995 for the calibration equation. A quality control check sample will be analyzed for every 20 samples; recovery must be within ±15 percent of the expected value.

353.3 - Nitrate is reduced to nitrite with hydrazine sulfate and the nitrite (that is originally present plus reduced nitrate) is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine dihydrochloride to form a highly colored azo dye which is measured colorimetrically.

Analysis should be made as soon as possible. If analysis can be made within 24 hours, samples should be preserved by refrigeration at 4°C. When samples must be stored for more than 24 hours, they should be preserved with 2 ml of sulfuric acid (H_2SO_4) per liter and refrigerated.

Calibration - A calibration curve will be generated using a reagent blank and three or more standards on a daily basis. The correlation coefficient must exceed 0.995 for the calibration equation. A quality control check sample will be analyzed for every 20 samples; recovery must be within ±15 percent of the expected value.

375.4 - Sulfate is a turbidimetric method where sulfate ion is precipitated as a barium sulfate suspension under controlled conditions. The resulting turbidity is determined by a nephelometer, filter photometer or spectrophotometer, and compared to a curve prepared from standard sulfate solutions.

Calibration - A calibration curve will be generated using a reagent blank and three or more standard sulfate solutions made from sulfuric acid. The correlation coefficient must exceed 0.995 for the calibration equation. A quality control check sample will be analyzed at a frequency of 10 percent; recovery must be within $\pm$10 percent of the expected value.

Quality Control Method Objectives

Table 8.1

GC Method SW8010

Parameter	Aqueous Matrix Accuracy (% Recovery)	Aqueous Matrix Precision (% RPD)	MDL (µg/L)	Solid Matrix Accuracy (% Recovery)	Solid Matrix Precision (% RPD)	MDL (mg/kg)
Bromodichloromethane	47 - 172 *	0 - 25 *	1.0	47 - 172 *	0 - 50 *	130
Bromoform	13 - 159 *	0 - 25 *	1.0	13 - 159 *	0 - 50 *	130
Carbon tetrachloride	43 - 143 *	0 - 25 *	1.0	43 - 143 *	0 - 50 *	130
Chlorobenzene	38 - 150 *	0 - 25 *	1.0	38 - 150 *	0 - 50 *	130
Chloroform	49 - 133 *	0 - 25 *	1.0	49 - 133 *	0 - 50 *	130
Dibromochloromethane	24 - 191 *	0 - 25 *	1.0	24 - 191 *	0 - 50 *	130
1,2-Dichlorobenzene	1 - 208 *	0 - 25 *	1.0	1 - 208 *	0 - 50 *	130
1,3-Dichlorobenzene	7 - 187 *	0 - 25 *	1.0	7 - 187 *	0 - 50 *	130
1,4-Dichlorobenzene	42 - 143 *	0 - 25 *	1.0	42 - 143 *	0 - 50 *	130
1,1-Dichloroethane	47 - 132 *	0 - 25 *	1.0	47 - 132 *	0 - 50 *	130
1,2-Dichloroethane	51 - 147 *	0 - 25 *	1.0	47 - 132 *	0 - 50 *	130
1,1-Dichloroethene	28 - 167 *	0 - 25 *	1.0	28 - 167 *	0 - 50 *	130
trans-1,2-Dichloroethene	38 - 155 *	0 - 25 *	1.0	38 - 155 *	0 - 50 *	13
1,2-Dichloropropane	44 - 156 *	0 - 25 *	1.0	44 - 156 *	0 - 50 *	1
cis-1,3-Dichloropropene	22 - 178 *	0 - 25 *	1.0	22 - 178 *	0 - 50 *	130
trans-1,3-Dichloropropene	22 - 178 *	0 - 25 *	1.0	22 - 128 *	0 - 50 *	130
Methylene Chloride	25 - 162 *	0 - 25 *	2.0	25 - 162 *	0 - 50 *	130
1,1,2,2-Tetrachloroethane	8 - 184 *	0 - 25 *	1.0	8 - 184 *	0 - 50 *	130
Tetrachloroethene	26 - 162 *	0 - 25 *	1.0	26 - 162 *	0 - 50 *	130
1,1,1-Trichloroethane	41 - 138 *	0 - 25 *	1.0	41 - 138 *	0 - 50 *	130
1,1,2-Trichloroethane	39 - 136 *	0 - 25 *	1.0	39 - 136 *	0 - 50 *	130
Trichloroethene	35 - 146 *	0 - 25 *	1.0	35 - 146 *	0 - 50 *	130
Trichlorofluoromethane	21 - 165 *	0 - 25 *	1.0	21 - 156 *	0 - 50 *	130

Quality Control Method Objectives

Table 8.2

GC Method SW8020

Parameter	Aqueous Matrix Accuracy (% Recovery)	Aqueous Matrix Precision (% RPD)	MDL (µg/L)	Solid Matrix Accuracy (% Recovery)	Solid Matrix Precision (% RPD)	MDL (mg/kg)
Benzene	62 - 155 *	0 - 25 *	0.5	62 - 155 *	0 - 25 *	65
Toluene	62 - 146 *	0 - 27 *	0.5	62 - 146 *	0 - 27 *	65
Ethylbenzene	61 - 155 *	0 - 23 *	0.5	61 - 155 *	0 - 23 *	65
m-p-Xylene	59 - 152 *	0 - 27 *	1.0	59 - 152 *	0 - 27 *	130
o-Xylene	60 - 149 *	0 - 25 *	1.0	60 - 149 *	0 - 25 *	130

Quality Control Method Objectives

Table 8.3

GC Method SW8040

	Aqueous Matrix		MDL	Solid Matrix		Matrix
	Accuracy	Precision		Accuracy	Precision	
Parameter	(% Recovery)	(% RPD)	(µg/L)	(% Recovery)	(% RPD)	(mg/kg)
4-Chloro-3-Methylphenol	50 - 122 *	0 - 25*	2.0	50 - 122 *	0 - 50*	70
2-Chlorophenol	38 - 126 *	0 - 25*	2.0	38 - 126 *	0 - 50*	70
4-Nitrophenol	13 - 110 *	0 - 25*	5.0	13 - 110 *	0 - 50*	170
Pentachlorophenol	36 - 134 *	0 - 25*	5.0	36 - 134 *	0 - 50*	170
Phenol	23 - 108 *	0 - 25*	2.0	23 - 108 *	0 - 50 *	70

* Denotes method QC limits.

Quality Control Method Objectives

Table 8.4

GC Method SW8080 and CLP-SOW

	Aqueous Matrix		MDL	Solid Matrix		Matrix
	Accuracy	Precision		Accuracy	Precision	
Parameter	(% Recovery)	(% RPD)	(µg/L)	(% Recovery)	(% RPD)	(mg/kg)
Aldrin	40 - 120 *	0 - 22*	.05	34 - 132 *	0 - 43 *	1.7
gamma-BHC (Lindane)	56 - 123 *	0 - 14*	.05	46 - 127 *	0 - 50 *	1.7
4,4'-DDT	38 - 127 *	0 - 27*	.10	23 - 134 *	0 - 50 *	3.3
Dieldrin	52 - 126 *	0 - 18*	.10	31 - 134 *	0 - 50 *	3.3
Endrin	56 - 121 *	0 - 21*	.10	42 - 139 *	0 - 38 *	3.3
Heptachlor	40 - 131 *	0 - 20*	.05	35 - 130 *	0 - 31 *	1.7

* Denotes method QC limits.
CLP-SOW 3/90

Quality Control Method Objectives

Table 8.5

GC Method SW8150

Parameter	Aqueous Matrix Accuracy (% Recovery)	Aqueous Matrix Precision (% RPD)	MDL (µg/L)	Solid Matrix Accuracy (% Recovery)	Solid Matrix Precision (% RPD)	Matrix (mg/kg)
2,4-D	40 - 150 *	0 - 25*	12.0	40 - 150 *	0 - 50 *	240
2,4,5-TP (Silvex)	40 - 150 *	0 - 25*	1.7	40 - 150 *	0 - 50 *	40
2,4,5-T	40 - 150 *	0 - 25*	2.0	40 - 150 *	0 - 50 *	34

* Denotes method QC limits.

Quality Control Method Objectives

Table 8.6

GC Method CA-DHS (mod)

	Aqueous Matrix		MDL	Solid Matrix		Matrix
	Accuracy	Precision		Accuracy	Precision	
Parameter	(% Recovery)	(% RPD)	(µg/L)	(% Recovery)	(% RPD)	(mg/kg)
Diesel	40 -150 *	0 - 25*	0.4	40 - 150 *	0 - 50 *	10

* Denotes method QC limits.

Quality Control Method Objectives

Table 8.7

GC Method SW8240 and CLP-SOW

	Aqueous Matrix		MDL	Solid Matrix		Matrix
	Accuracy	Precision		Accuracy	Precision	
Parameter	(% Recovery)	(% RPD)	(µg/L)	(% Recovery)	(% RPD)	(mg/kg)
1,1- Dichloromethane	61 - 145 *	0 - 14*	10	59 - 172 *	0 - 22	10
Trichloromethane	71 - 120 *	0 - 14*	10	62 - 137 *	0 - 24	10
Benzene	76 - 127 *	0 - 11*	10	66 - 142 *	0 - 21	10
Chlorobenzene	75 - 130 *	0 - 13*	10	60 - 133 *	0 - 21	10
Toluene	76 - 125 *	0 - 13*	10	59 - 139 *	0 - 21	10

* Denotes method QC limits.

Quality Control Method Objectives

Table 8.8

GC/MS Method SW8270 and CLP-SOW

	Aqueous Matrix		MDL	Solid Matrix		Matrix
	Accuracy	Precision		Accuracy	Precision	
Parameter	(% Recovery)	(% RPD)	(µg/L)	(% Recovery)	(% RPD)	(mg/kg)
Phenol	12 - 110 *	0 - 42*	10	26 - 90 *	0 - 35*	330
2-Chlorophenol	27 - 123 *	0 - 40*	10	25 - 102 *	0 - 50*	330
1,4- Dichlorobenzene	36 - 97 *	0 - 28*	10	28 - 104 *	0 - 27*	330
N-Nitroso-di-n-propylamine	41 - 116 *	0 - 38*	10	41 - 126 *	0 - 38*	330
1,2,4-Trichlorobenzene	39 - 98 *	0 - 28*	10	38 - 107 *	0 - 23*	330
4-Chloro-3-methylphenol	23 - 97 *	0 - 42*	10	26 - 103 *	0 - 33*	330
Acenaphthene	46 - 118 *	0 - 31*	10	31 - 137 *	0 - 19*	330
4-Nitrophenol	10 - 80 *	0 - 50*	25	11 - 114 *	0 - 50*	800
2,4-Dinitrotoluene	24 - 96 *	0 - 38*	10	28 - 89 *	0 - 47*	330
Pentachlorophenol	9 - 103 *	0 - 50*	25	17 - 109 *	0 - 47*	800
Pyrene	26 - 127 *	0 - 31*	10	35 - 142 *	0 - 36*	330

* Denotes method QC limits.

Quality Control Method Objectives

Table 8.9

Metals Method SW6010, SW7060, SW7421, SW7740, SW7470, SW7841, and CLP-SOW

	Aqueous Matrix		MDL	Solid Matrix		Matrix
	Accuracy	Precision		Accuracy	Precision	
Parameter	(% Recovery)	(% RPD)	(µg/L)	(% Recovery)	(% RPD)	(mg/kg)
Aluminum	75 - 125 *	0 - 20*	200.0	75 - 125 *	0 - 20*	40.00
Antimony	75 - 125 *	0 - 20*	60.0	75 - 125 *	0 - 20*	12.00
Arsenic	75 - 125 *	0 - 20*	10.0	75 - 125 *	0 - 20*	2.00
Barium	75 - 125 *	0 - 20*	200.0	75 - 125 *	0 - 20*	40.00
Cadmium	75 - 125 *	0 - 20*	5.0	75 - 125 *	0 - 20*	1.00
Calcium	75 - 125 *	0 - 20*	5000.0	75 - 125 *	0 - 20*	1,000.00
Chromium	75 - 125 *	0 - 20*	10.0	75 - 125 *	0 - 20*	2.00
Cobalt	75 - 125 *	0 - 20*	50.0	75 - 125 *	0 - 20*	10.00
Copper	75 - 125 *	0 - 20*	25.0	75 - 125 *	0 - 20*	5.00
Iron	75 - 125 *	0 - 20*	100.0	75 - 125 *	0 - 20*	20.00
Lead	75 - 125 *	0 - 20*	3.0	75 - 125 *	0 - 20*	0.60
Magnesium	75 - 125 *	0 - 20*	5000.0	75 - 125 *	0 - 20*	1,000.00
Manganese	75 - 125 *	0 - 20*	15.0	75 - 125 *	0 - 20*	3.00
Mercury	75 - 125 *	0 - 20*	0.2	75 - 125 *	0 - 20*	0.04
Nickel	75 - 125 *	0 - 20*	40.0	75 - 125 *	0 - 20*	8.00
Potassium	75 - 125 *	0 - 20*	5000.0	75 - 125 *	0 - 20*	1,000.00
Selenium	75 - 125 *	0 - 20*	5.0	75 - 125 *	0 - 20*	1.00
Silver	75 - 125 *	0 - 20*	10.0	75 - 125 *	0 - 20*	2.00
Sodium	75 - 125 *	0 - 20*	5000.0	75 - 125 *	0 - 20*	1,000.00
Thallium	75 - 125 *	0 - 20*	10.0	75 - 125 *	0 - 20*	2.00
Vanadium	75 - 125 *	0 - 20*	50.0	75 - 125 *	0 - 20*	10.00
Zinc	75 - 125 *	0 - 20*	20.0	75 - 125 *	0 - 20*	4.00
Cyanide	75 - 125 *	0 - 20*	10.0	75 - 125 *	0 - 20*	0.00

* Denotes method QC limits.

Quality Control Method Objectives

Table 8.10

Water Quality Parameters

	Aqueous Matrix		MDL	Solid Matrix		Matrix
	Accuracy	Precision		Accuracy	Precision	
Parameter	(% Recovery)	(% RPD)	(µg/L)	(% Recovery)	(% RPD)	(mg/kg)
Acidity		0 - 20*	10.0		0 - 20*	NA
Alkalinity		0 - 20*	10.0		0 - 20*	NA
BOD-5day		0 - 20*	2.0		0 - 20*	NA
Bromide	75 - 125 *	0 - 20*	2.0		0 - 20*	NA
Chlorine-Resid		0 - 20*	0.5		0 - 20*	2.0
CN-CLP	75 - 125 *	0 - 20*	.02	75 - 125 *	0 - 20*	1.0
CN-Free		0 - 20*	.03		0 - 20*	1.0
CN-SW	75 - 125 *	0 - 20*	.02	75 - 125 *	0 - 20*	1.0
CN-TOT	75 - 125 *	0 - 20*	.02			1.0
COD	75 - 125 *	0 - 20*	10.0			NA
Color		0 - 20*	5.0			NA
Hexavalent Chromium	75 - 125 *	0 - 20*	.5	75 - 125 *	0 - 20*	5.0
F-Distillation	75 - 125 *	0 - 20*	.1	75 - 125 *	0 - 20*	1.2
Hardness		0 - 20*	10.0			NA
Ignitability		0 - 20*	NA		0 - 20*	NA
Iodide	75 - 125 *	0 - 20*	2.0			NA
Moisture		0 - 20*	NA		0 - 20*	NA
Moisture-Solv		0 - 20*	NA		0 - 20*	NA
Ammonia-Dist	75 - 125 *	0 - 20*	.05	75 - 125 *	0 - 20*	2.0
Nitrate	75 - 125 *	0 - 20*	.05	75 - 125 *	0 - 20*	1.0
Org Nitrogen	75 - 125 *	0 - 20*	.05	75 - 125 *		2.0
TKN	75 - 125 *	0 - 20*	.05	75 - 125 *	0 - 20*	2.0
Odor		0 - 20*	1.0			NA
Dissolved Oxygen		0 - 20*	.05			NA
pH		0 - 20*	NA		0 - 20*	NA
Hydrolyzable Phosphorus	75 - 125 *	0 - 20*	.01	75 - 125 *	0 - 20*	.1
Ortho Phosphorus	75 - 125 *	0 - 20*	.05	75 - 125 *	0 - 20*	.1
Total Phosphorus	75 - 125 *	0 - 20*	.05	75 - 125 *	0 - 20*	.1
Reactive Sulfide	75 - 125 *	0 - 20*	1.0	75 - 125 *	0 - 20*	2.0
Reactive Cyanide	75 - 125 *	0 - 20*	.02	75 - 125 *	0 - 20*	1.0
Phenols	75 - 125 *	0 - 20*	.05	75 - 125 *	0 - 20*	2.5
Oil & Grease		0 - 20*	5.0		0 - 20*	50.0
Sulfate	75 - 125 *	0 - 20*	1.0	75 - 125 *	0 - 20*	10
Sulfide	75 - 125 *	0 - 20*	1.0	75 - 125 *	0 - 20*	20

Quality Control Method Objectives

Table 8.10

Water Quality Parameters

	Aqueous Matrix		MDL	Solid Matrix		Matrix
	Accuracy	Precision		Accuracy	Precision	
Parameter	(% Recovery)	(% RPD)	(µg/L)	(% Recovery)	(% RPD)	(mg/kg)
Sulfite	75 - 125 *	0 - 20*	2.0			NA
TSS		0 - 20*	4.0			NA
TDS		0 - 20*	0.2			NA
Total Solids		0 - 20*	10.0			NA
Total Volatile Solids		0 - 20*	10.0			NA
Specific Conductance		0 - 20*	1.0			1.0
Surfactants	75 - 125 *	0 - 20*	.025			NA
TOX		0 - 20*	.01			NA
Turbidity		0 - 20*	.1			NA
TOC	75 - 125 *	0 - 20*	1.0	50 - 150 *	0 - 20*	100
TPH-IR	75 - 125 *	0 - 20*	.1			NA
Chloride	75 - 125 *	0 - 20*	5.0	50 - 150 *	0 - 20*	NA
Nitrite	75 - 125 *	0 - 20*	.05	75 - 125 *	0 - 20*	1.0

* Denotes method QC limits.

SECTION 9

CALIBRATION PROCEDURES AND FREQUENCY

Currently, PACE, Inc. combines analytical method descriptions with the procedure for calibration and frequency. In the near future, in order to comply with certain state formats, PACE, Inc. will separate the above information to be provided in this section.

SECTION 10

PREVENTATIVE MAINTENANCE

10.0 Introduction

The primary objective of a preventative maintenance program is to help ensure the timely and effective completion of a measurement effort. PACE, Inc.'s preventative maintenance program is designed to minimize the down time of crucial sampling and/or analytical equipment due to expected or unexpected component failure. In implementing this program, efforts are focused in three primary areas.

- Establishment of maintenance responsibilities if there is not a service contract.
- Establishment of maintenance schedules for major and/or critical instrumentation and apparatus that are subject to wear, deterioration, or other change in operational characteristics without periodic maintenance.
- Establishment of an adequate inventory of critical spare parts and equipment that should be available within the laboratory to minimize downtime.

10.1 Maintenance Responsibility

Maintenance responsibilities for permanently assigned equipment are assigned to the respective laboratory managers. The laboratory managers then establish maintenance procedures and schedules for each major equipment item. Responsibilities for specific items may be delegated to laboratory personnel, although the laboratory managers retain responsibility for ensuring adherence to prescribed protocol. In most cases, this responsibility is passed to the manufacturer since most instruments are under service contracts.

10.2 Maintenance Schedules

The effectiveness of any maintenance program depends to a large extent on adherence to specific maintenance schedules for each major equipment item. A specific schedule is established for all routine maintenance activities. Other maintenance activities may also be identified as requiring attention on an as-needed basis. Manufacturers' recommendations provide the primary basis for the established maintenance schedules, and manufacturers' service contracts provide primary maintenance for many major instruments (e.g. GC instruments, atomic absorption spectrometers, analytical balances, etc.). All aspects of routine and non-routine instrument maintenance are recorded in logbooks, and a logbook is dedicated to each instrument.

10.3 Spare Parts

Along with a schedule for maintenance activities, an adequate inventory of spare parts is required to minimize equipment downtime. This inventory should emphasize those parts and supplies which:

- are subject to frequent failure;
- have limited useful lifetimes;
- cannot be obtained in a timely manner should failure occur.

Laboratory supervisors are responsible for maintaining an adequate inventory of necessary spare parts.

10.4 Maintenance Contracts

All major pieces of analytical equipment are under service contracts with the manufacturers. These maintenance contracts provide for preventative maintenance as well as spare parts inventory recommendations and service in the case of equipment failure.

SECTION 11

INTERNAL QUALITY CONTROL

11.0 Introduction

An internal quality control system is a set of routine internal procedures for assuring that the data output of a measurement system meets prescribed criteria for data quality. Inherent and implied in this control function is a parallel function of measuring and defining the quality of the data output. A well- designed internal QC program must be capable of controlling and measuring the quality of the data, in terms of precision and accuracy. Precision reflects the influence of the inherent variability in any measurement system. Accuracy reflects the degree to which the measured value represents the actual or "true" value for a given parameter, and includes elements of both bias and precision. Accuracy of measurement data is related to the precision and bias of the component parts of the measurement system.

Generally, internal quality control procedures may be divided into two overlapping categories. One category includes those procedures which are used to control data quality within prescribed limits of acceptability. These acceptability limits are usually related to data precision, accuracy, and completeness. The other category includes those procedures designed to provide a quantitative assessment of data quality, again in terms of precision, accuracy, and completeness. Some internal QC procedures, by their nature, serve both control and assessment functions.

This section addresses QC procedures associated with analytical efforts. Included are general quality control considerations, as well as specific quality control checks which provide ongoing control and assessment of data quality, in terms of precision and accuracy. Quality control checks which provide the basis for quantitative control and assessment of data quality, along with required frequency, acceptance criteria, and corrective action, are discussed in detail in other sections of this QAP. Although outside the scope of this

document, a brief discussion of sampling QC is presented below. The use of control charts is discussed later in this section.

11.1 Sampling QC

Quality control procedures should be an integral part of each sampling methodology, and should include procedures which will ensure the collection of representative samples which are free from external contamination. Although different extraction and/or analytical procedures will be used for the various parameters of interest, certain general quality control procedures are applicable to most sampling methods. These include the following:

11.1.1 Field Blanks

Field blanks should be collected routinely, at a minimum frequency of one per 20 samples (5%). These will be sample containers (jars and vials) which are handled (i.e., opened, sealed, and transported) in the same manner as actual sample containers. When feasible, reagent grade sand will be loaded into the sample containers and submitted for analysis. A least one field blank will be taken for each type of sample taken during the course of a specific sampling program.

11.1.2 Matrix Spike/Matrix Spike Duplicate

Split samples for matrix spike and matrix spike duplicate samples should be collected at a frequency of 5% (1 set per 20 samples) to provide a measure of method variability (i.e., total variability due to imprecision in both sampling and analytical procedures). At least one MS/MSD sample should be taken for each type of sample taken over the course of a specific sampling period.

11.1.3 Chain-of-Custody

Chain-of-custody forms should accompany all samples.

11.1.4 Cleaning

Sampling apparatus should be thoroughly cleaned between each sampling to prevent cross-contamination of the samples.

In addition to these general sampling QC requirements, additional QC procedures should be performed as part of the analytical methods. These are discussed below in the following subsections.

11.2 Laboratory QC

Laboratory QC Components definitions and usages are described in Section 5. Dependent upon the methods performed and the requirements of the specific project, the appropriate QC Components, criteria and protocols are followed by the laboratory. For each "project" requiring such a unique QAP may be prepared which identifies the specific QC information under which the laboratory will perform.

11.3 Establishment of Laboratory Control Limits

The laboratory has a quality control program in place designed to minimize random errors, monitor accuracy, monitor precision, and flag emerging systematic problems. This is demonstrated through the use of Quality Action Logs and performance evaluation samples and the corrective actions that are implemented as a result of their findings.

Action limits for quality control samples are used to indicate variability in results due to systematic or assignable as opposed to random or unassignable causes. For most projects, the control limits and required corrective action or documentation are those set forth by the USEPA CLP SOW for Inorganic (7/88 or 3/90) and Organic (2/88 or 3/90) analyses.

11.3.1 Controls Charts

Based upon the empirical data collected within the laboratory for each method, for each QC component, the QA Officer will maintain a database to aid in the determination of laboratory performance in terms of precision and accuracy, as well as determine the statistical control and warning limits. At a minimum, laboratory defined statistics will be prepared yearly with control charts. For many projects or programs needs (e.g., NAVY, NEESA, ARMY CORPS, etc.) the specific requirements for control charting and reporting will be performed by the laboratory.

Data and Quality Control Charts are reviewed by the QA Officer as they are prepared, in conjunction with the appropriate laboratory department manager to ensure there is an understanding of the data. If there are any concerns or potential investigations requested they will be initiated by the QA Officer.

11.4 Precision

The precision of analytical laboratory data can be evaluated using (1) standard deviation, (2) range, (3) coefficient of variation, also known as the relative standard deviation, and (4) percent difference.

(1) The standard deviation is a measure of the average distance of individual observations from the mean. It is usually denoted *s* and defined as:

$$\sqrt{\frac{\sum_{i=1}^{n}(X_i-X)^2}{n-1}} \quad \text{or} \quad \sqrt{\frac{\sum_{i=1}^{n}X_i^2 - \left(\sum_{i=1}^{n}X_i\right)^2/n}{n-1}}$$

In these equations, n is the sample size,
X_i is the i^{th} observation in the sample, and
X is the sample mean.

(2) The range is the largest observation in a data set minus the smallest observation in the data set, often denoted as *R*.

(3) The coefficient of variation (CV), or relative standard deviation, is a commonly-used measure of variability that is adjusted for the magnitude of the values in the sample:

$$CV = \frac{\text{Standard Deviation}}{\text{Mean}} \times 100$$

The coefficient of variation is used most often when the size of the standard deviation changes with the size of the mean. When individual measurements of CV or standard deviation are combined (pooled) to obtain an overall measure of variability for a given type of analysis for measurement, the following technique may be used:

$$\text{Pooled CV} = \sqrt{\frac{\sum_{i=1}^{n} (X_i^2 DF_1)}{\sum_{i=1}^{n} DF_1}}$$

In this equation, X_1 = CV of data set i (e.g. CV for one duplicate pair *i*);
DF_1 = degrees of freedom from data set *i* (k_i - 1);
n = total number of data sets (e.g. total number of duplicate pairs);
k_1 = number of data points in set *i* (e.g. k=2 for duplicates); and
i = data set 1,2,3 ... n.

(4) Relative Percent Difference (RPD) is another commonly-used measure of variability that is adjusted for the magnitude of the measured values. It is used only when the sample contains only two observations and is given by:

$$\text{RPD} = \frac{(X_1 - X_2)}{\frac{(X_1 + X_2)}{2}} \times 100$$

where X_1 and X_2 are duplicate sample measurement results.

11.5 Accuracy

The accuracy of analytical laboratory data is usually presented in terms of (1) relative error and (2) confidence intervals at the 95% level.

(1) Percent Relative Error = $\frac{\text{Measured Value - Actual Value}}{\text{Actual Value}} \times 100\%$

(2) 95% Confidence Interval = $X \pm {}^{t}(a, n-1)$

In this equation, X = sample mean;
s = sample standard deviation;
n = sample size;
a = risk level (0.025 for the 95% confidence interval);
$t(a, n-1)$ = value of the tabulated student's "t" distribution for n-1 degrees of freedom and risk level *a*.

Accuracy may also be expressed as percent recovery, given by:

$$\% \text{ Recovery} = \frac{\text{Measured Value}}{\text{Actual Value}} \times 100\%$$

Percent recover is related to percent relative error by:

$$\% \text{ Recovery} = \% \text{ Relative Error} + 100$$

Spike recover is commonly used to determine the performance of a method:

$$\% \text{ spike recovery} = \frac{\text{value of sample} + \text{added spike} - \text{value of unspiked sample}}{\text{value of spike added}}$$

SECTION 12

DATA REDUCTION, EVALUATION AND REPORTING

Data presentation begins with processing data and continues through the review and reporting of analytical results. The following steps shall be used to evaluate all data:

12.1 Data Reduction

- Initial Review - The analyst who performed the analysis shall be responsible for reviewing all data, calculations and results. Each item and/or calculation reviewed shall be marked with a checkmark and the bottom right-hand corner of each page is to be initialed and dated, indicating that the review of that page is complete.

- Peer Review - For any data which are complicated and/or the analyst would like a secondary review, the data may be submitted to a second member of the department for a peer review prior to a manager review.

- Final Review - The Laboratory Managers/Supervisors shall be responsible for reviewing all data for completeness and to ensure that all associated Quality Control data has been verified. The Managers/Supervisors shall initial and date each page of the analytical results. The data package is then turned into the Report Center Department.

- Review of Data Reporting - In the Report Center Department, the draft data report is prepared and the reported results checked against the reviewed processed data so that transcription errors do not occur. Using the draft report, all data entries shall be checked. The checker is under the supervision of the Report Center Manager. As each item on the draft report is checked, a checkmark will be placed beside each correct entry. Corrections shall be made using one line through the incorrect entry, with the correct entry written beside it. The checker shall indicate that the corrections have been made by placing a second checkmark next to the correction, after comparing to the revised data report. The checker shall initial and date each page of the draft report.

- Final Approval - Once the report is complete, the Report Center Manager will conduct an inspection of the entire package to assure completeness. This review is documented on the Report file jacket.

In addition to the above data evaluation procedure, the Quality Assurance Officer, or General Manager or designate, shall review a minimum of 10% of the completed data packages to ensure that the data evaluation procedure is being followed and that appropriate narrative discussing the anomalies or noncomformances, and corrective actions, has been prepared. The cover letter or narrative shall be signed by the QA Officer, Report Center Manager, or the Analytical Department Manager.

12.2 Data Evaluation (Validation)

As discussed in Section 12.1, the review of all technical data and subsequent acceptance or rejection of data is based upon the specific Quality Control and Quality Assurance Criteria as contained within the methodology SOPs. Typically the QC components evaluated for each sample and/or sample batch include instrument calibration and performance method blanks, continuing calibration blanks and standards, sample duplicates and spikes, external lab control sample analyses, and surrogate and/or internal standard recoveries. The specific criteria for data acceptance, flagging, qualification, etc. as defined with the method SOP utilized.

12.3 Data Reporting

The format and content of a data report is dependent upon project needs, such as whether or not an explanatory test is required, client or contract requirements, and government agency reporting formats. The PACE, Inc. Quality Assurance Program does not specify a report format; however, the following are applicable to data presentation.

- Data are presented in a tabular format whenever possible.
- Each data package is identified with the project number and name; date of issue; and client name.

- Data presentation includes:
  - Sample identification number used by the PACE, Inc. laboratory and/or the sample identification provided to the laboratory, if different from identification used in the laboratory;
  - Chemical parameters analyzed, reported values, and units of measurement;
  - Detection limit of the analytical procedure if the reported value is less than the detection limit;
  - Data for a chemical parameter are reported with consistent significant figures for all samples;
  - Date sampled, date received, date prepared/extracted, date analyzed;
  - Results of Quality Control sample analysis, if appropriate;
  - Achieved accuracy, precision, and completeness of data, if appropriate;
  - Footnotes referenced to specific data if required to explain reported values;
  - Qualifications and/or flags to the data where appropriate by either method used or client requirement, in accordance with the method utilized for analyses (i.e. CLP SOW).
- Data should be transmitted from the laboratory only upon approval of the Laboratory Manager or Laboratory Director if preliminary or verbal data requested.

12.4 Data Archival

The purpose of the Records Management System is to standardize the organization, storage and retrieval of all data and documents pertinent to quality and the analytical process. To fulfill these requirements, PACE, Inc. maintains a Records Management System which meets the following criteria.

- Data and documents are easily retrievable.
- Files are secure.
- Data and documents are indexed.
- A formal document inventory can be produced if required by the contract/project.
- Project-specific data documents are grouped together.
- Laboratory Operation/QA Documents are cross-referenced to applicable projects.
- The system is documented in the Quality Assurance Manual and Standard Operating Procedures.
- There is a designated "File Custodian" responsible for the Project and Lab Operation/QA file maintenance.
- Specific regulatory or contractual requirements can be accommodated.

SECTION 13

CORRECTIVE ACTION

Internal and external corrective action issues for data quality are addressed through Quality Action (QA) logs. The function of a QA log is to document questions or concerns regarding any aspect of our laboratories operations: data, procedures, QA or QC.

The evaluation of QA logs will provide service to our clients and allow us to clarify or correct any non-conformances or errors made in our operation. After the QA log has been resolved for the specific instance, if the QA log investigation has uncovered a systematic error or a need for method procedure clarify action, a QA memorandum is issued by the QA office to the department managers. QA memorandums are then incorporated into SOP inserts or modifications in a timely manner.

13.1 QA Log Tracking

A corrective action issue may be raised by a client, auditor, or any PACE, Inc. employee. QA logs will be issued only by the QA office unless methodologies mandate re-extraction or re-digestion of a sample or sample batch. QA logs regarding these matters are issued by the department manager to the appropriate department manager, and the original is given to the QA office.

The QA office is responsible for the management (and maintaining) of QA logs. each department is responsible for the resolution of a QA log. The QA office records the log in a computer spreadsheet to track response time and gather information on error frequency.

All resolved QA logs will be returned to the QA office for final review. Once the QA log is reviewed and found adequate, the QA office will not the final resolution on the original QA log. The QA office is responsible for seeing that the final resolution is carried out, whether it is in a report resubmission, a client phone call, or a QA memorandum.

All changes to data or reports made after reports have been issued must be documented with a QA log. Reports will be re-issued or clients contacted with the consent of the QA officer.

Completed QA logs will be so marked and filed in the QA office.

13.2 AR Tracking

13.3 Corrective Action Follow-up

Each month a summary of all QA logs for that month are printed and reviewed by the QA officer and General Manager to determine if there are any systematic problems in the laboratory's operation. If any further investigations or changes are warranted, they will be initiated by the QA officer.

SECTION 14

PERFORMANCE AND SYSTEM AUDITS

The purpose of a Quality Assurance Audit is to provide an objective, independent assessment of a measurement effort. It ensures that the laboratory's data-generating, data-gathering, and measurement activities produce reliable and useful results.

Quality assurance audits play an important role in PACE, Inc.'s overall QA/QC Program. This section describes the role of the QA Auditor and the nature of quality assurance audits.

14.1 Quality Assurance Auditor

The QA Auditor is the person who designs and/or performs QA performance and systems audits. Since QA audits represent, by definition, independent assessments of a measurement system and associated data quality, the auditor must be functionally independent of the measurement effort to ensure objectivity. However, the auditor must be familiar enough with the objectives, principles, and procedures of the measurement efforts to be able to perform a thorough and effective evaluation of the measurement system. Especially important is the ability to identify components of the system that are critical to overall data quality. For this reason, the audit focuses heavily upon those elements.

PACE, Inc.'s organizational structure, described in Section 3, ensures the independence of the QA office. A QA Designee is given the responsibility for management and execution of audit activities. The QA Designee is responsible for designing and performing both technical systems and data quality audits.

14.2 Technical Systems Audits

A technical systems audit is an on-site, qualitative review of the various aspects of a total sampling and/or analytical system. It is an assessment of overall effectiveness. It represents an objective and insightful evaluation of a set of interactive systems with respect to strengths, deficiencies, and potential areas of concern. Typically, the audit consists of observations and documentation of all aspects of the measurement effort.

Annually, the QA Office will conduct a technical systems audit of a particular department. The audit will cover, but is not limited to:

1) Sample receipt

2) Sample (extract) storage

3) Sample preparation and cleanup

4) Equipment checks

5) Instrument calibration and maintenance

6) Sample analyses

7) Sample disposal

8) Logbooks

9) SOPs pertaining to all mentioned above

Technical Systems Audits do not answer quantitative questions about the measurement system. The Technical Systems Audits will be conducted at a minimum of once yearly or more frequently when deemed necessary by the QA Officer.

14.3 Audits for Data Quality

The purpose of audits for data quality is to assess data quality indicators. Audits for data quality provide information required to characterize data quality by answering questions. Annually, the QA office will conduct a data quality audit of a particular department. Often this will coincide with the department's technical systems audit. This audit will cover but is not limited to:

1) Data generation

2) Data reduction

3) Data calculation

4) Data review

5) Data reporting

6) Data backup

7) Archive

8) Training documentation

9) SOPs pertaining to all mentioned above

Audits of data quality answer questions of whether the data collection efforts need modifications, and whether the use and documentation of quality control procedures are adequate. Audits of data quality do not, however, answer technical questions such as those concerning the operating conditions of facilities and equipment.

14.4 Post-Audit Debriefing

Following each audit, a post-audit debriefing session is conducted. The purpose of this session is to discuss preliminary audit results with the audit participants. If the audit reveals a critical deficiency, an "QA Action Report" will be issued (see Section 12 of this manual). The debriefing session is followed by a detailed audit report that identifies areas of concern and recommendations for corrective actions. These audit reports and documented actions will be kept in QA file.

SECTION 15

REFERENCES AND RESUMES

PACE, Inc. currently participates in the U.S. EPA's Contract Laboratory Program (CLP) and has sustained its participation in this program since 1983. PACE, Inc. is currently the only active CLP participant in the Gulf Coast area.

In addition, PACE, Inc. is routinely requested by EPA to participate in the CLP's Special Analytical Services (SAS) Program for both organics and inorganics. In order to participate in SAS, CLP labs must qualify in the areas of data quality, completeness, and on-time performance in the routine program.

PACE, Inc. is also certified by the states of New Jersey, California and Connecticut. Florida certification has been applied for and is only awaiting the review and approval of our QAP. Other certifications include the Army Corps of Engineers and Naval Energy and Environmental Support Activities.

PACE, Inc. also routinely partakes in many outside performance evaluation study, such as EMSL/Cincinnati Water Pollution Study and EPA CLP Quarterly Blinds.

PACE, Inc. has submitted the resumes of its key personnel in this section of the Quality Assurance Plan.

HARRY J. KLANN

POSITION	Vice President, General Manager and Laboratory Director, ETC/Gulf South, New Orleans, LA.
EDUCATION	Central Michigan University, M.S., Environmental Science, graduated with honors; teaching assistantship; 1976.
	Michigan Technological University, B.S., Biological Science, graduated with honors, 1973.
PROFESSIONAL AFFFILIATIONS	American Chemical Society American Society for Quality Control
PROFESSIONAL EXPERIENCE	Vice President, General Manager and Laboratory Director, ETC/Gulf South, New Orleans, Louisiana, 1989 - present.
	Technical Marketing Specialist, Director of Quality Assurance, Laboratory Manager, Production/Technical Services Manager, Quality Assurance Project Officer, Data Management Supervisor for ETC, 1983 - 1989.
	Quality Assurance Supervisor, Laboratory Coordinator, Senior Field/Laboratory Scientist, Lawler, Matusky & Skelly Engineers (LMS), 1977 - 1983.
	Field and Laboratory Supervisor, Limnological Services at Central Michigan University, 1976 -1977.

EXPERIENCE SUMMARY

Mr. Klann is the Vice President, General Manager and Laboratory Director of ETC/Gulf South. He has over sixteen years of experience in the environmental analytical field. Mr. Klann is responsible for directing all technical and administrative functions of the laboratory. He is responsible for the overall management of the laboratory and the timely production of technically sound data. He acts as Project Director for specific projects.

Mr. Klann was previously the Technical Marketing Specialist for ETC in Edison, New Jersey. As such, he was responsible for providing technical and quality assurance expertise and support to marketing, laboratory staff and ETC clients. This expertise and support included technical project preparation and/or evaluation and meeting with clients, consultants and regulatory personnel with regard to environmental regulations, analytical chemistry, quality control/assurance, laboratory operations, data defensibility and interpretation.

Mr. Klann held several other positions at ETC, including Director of Quality Assurance, Laboratory Manager, Production/Technical Services Manager, Quality Assurance Project Officer and Data Management Supervisor. His responsibilities included developing, implementing and managing network's comprehensive QA/QC program, training and managing QA/QC department staff, meeting all applicable federal and state regulations and/or contract requirements and supervising technical groups and non laboratory production departments.

At Lawler, Matusky & Skelly Engineers, Mr. Klann held the positions of Senior Field/Laboratory Scientist, Laboratory Coordinator and Quality Assurance Supervisor. His responsibilities included coordinating the performance of aquatic studies for Consumers Power Company's nuclear power plant site at Midland, Michigan; providing work hour and budget projections, review and implementation of technical procedures to improve efficiency and quality of analyses; and management of the QA/QC activities of all laboratory, field and data efforts for five laboratories.

Early in his career, Mr. Klann supervised the technical staff for the preoperational aquatic assessment of Consumers Power Company's nuclear plant site for Limnological Services at Central Michigan University.

WILLIAM R. DECKELMANN

POSITION	Client Services Manager, ETC/Gulf South, New Orleans, LA.
EDUCATION	Master of Science, Microbiology, 1987 Long Island University, Brooklyn, New York
	Bachelor of Science, Biology, 1981 Harpur College, Binghamton, New York
PROFESSIONAL EXPERIENCE	Client Services Manager ETC/Gulf South, New Orleans, LA, 12/90 - Present.
	Manager of Work Group and Sample Management Environmental Testing and Certification Corporation, Edison, N.J. 4/87 - 12/90
	Quality Control/Assurance Supervisor, Turner Hall Corporation, Newburg, NY 1985 - 1987
	Quality Control Supervisor, Diagnostic Reagent Tech, Inc., Teaneck, N.J. 1984 - 1985
	Chemist, Becton Dickinson Immunodiagnostics, Orangeburg, N.Y. 1981 - 1984

EXPERIENCE SUMMARY

Mr. Deckelmann currently manages the Client Services and Sample Management Departments at ETC/Gulf South. This involves extensive coordination with our clients, sales staff and technical personnel to ensure new business continues to flow in and quality sample data is produced on time.

Prior to joining ETC/Gulf South, Mr. Deckelmann managed a Work Group and Sample Management Department at Environmental Testing and Certification Corporation. His responsibilities included project management, resolving technical issues and directing laboratory operations.

While at Turner Hall Corporation, Mr. Deckelmann performed as Quality Control/Assurance Supervisor which involved ensuring company regulatory compliance, site inspection and following QA process through completion of finished product.

At Diagnostic Reagent Tech., Inc., his Quality Control Supervisor duties encompassed development of a Q.C. program for the manufacture of diagnostic products.

As a Chemist at Becton Dickinson Immunodiagnostics, Mr. Deckelmann was responsible for all aspects of large scale production of Radio-immunoassay diagnostic products.

ELAINE A. WILD

POSITION	Quality Assurance Officer, ETC/Gulf South, New Orleans, LA.
EDUCATION	University of New Orleans, B.S., Chemistry, 1988.
PROFESSIONAL EXPERIENCE	Quality Assurance Officer, ETC/Gulf South, New Orleans, LA, New Orleans, LA, 1992 - present.
	Chemist, Gas Chromatography Group, Gulf South Environmental Laboratory, Inc., New Orleans, LA, 1989 - 1992.
	Lab Technician, Water Quality Laboratory, Gulf South Environmental Laboratory, Inc., New Orleans, LA, 1987 - 1988.

EXPERIENCE SUMMARY

As Quality Assurance Officer, Ms. Wild is responsible for the administration of the laboratory's QC and QA programs. These include SOP's, PE's, internal audits and QC samples, and our corrective action procedures.

As a Chemist at Gulf South Environmental Laboratory, Inc., Ms. Wild was responsible for analyzing environmental samples using Hewlett-Packard's 5890 Gas Chromatograph. Additional responsibilities include requisitioning and receiving of chemical supplies and equipment and running gas chromatography analysis by EPA protocol on soil and waste water samples.

As a Lab Technician in the Water Quality Laboratory, she was responsible for performing EPA approved methods for solid, sludge, waste water and drinking water parameters as specified by NPDES and RCRA protocols. She has also performed water quality analysis on various environmental matrices and for bio-monitoring facilities.

SHELLEY R. ANTOINE

POSITION	Manager, GC/MS Laboratory, ETC/Gulf South, New Orleans, Louisiana.
EDUCATION	B.A. in Biological Sciences, University of New Orleans, New Orleans, Louisiana, 1976.
	M.S. in Biological Sciences, University of New Orleans, New Orleans, Louisiana, 1984.
PROFESSIONAL EXPERIENCE	Manager, GC/MS Laboratory, ETC/Gulf South, New Orleans, Louisiana, June, 1990-present.
	Supervisor, Volatile Organics GC/MS Laboratory, ETC/Gulf South, New Orleans, Louisiana, 1988-1990.
	GC/MS Operator, ETC/Gulf South, New Orleans, Louisiana, 1987-1988.
	Task Leader (Research Associate), Center for Bio-Organic Studies, University of New Orleans, New Orleans, Louisiana, 1980-1987.
	Research Associate, Center for Bio-Organic Studies, University of New Orleans, New Orleans, Louisiana, 1976-1980.

EXPERIENCE SUMMARY

Ms. Antoine is the GC/MS Laboratory Manager. She has a Master's of Science in Biology and over fifteen years of experience in gas chromatography and GC/MS. As Lab Manager, Ms. Antoine is responsible for all aspects of the GC/MS laboratory operation including management of the work flow, work assignments for personnel, final data review, instrument maintenance and the implementation and oversite of the quality control program for the laboratory. She is also responsible for the recruitment and training of new analysts.

Before coming to ETC/Gulf South, Ms. Antoine was task leader and research associate at the Center for Bio-Organic Studies, University of New Orleans working on a wide variety of programs funded by various government agencies and private industries; she was responsible for operation and routine maintenance of a Hewlett-Packard 5985 GC/MS/Data System and performed GC/MS data treatment on a Finnigan INCOS 2000 Series Data System with user-prepared programs and standard Finnigan software.

Ms. Antoine has conducted GC and GC/MS analysis of environmental, biological, and industrial samples for volatile organics, petroleum hydrocarbons, chlorinated organics, pesticides, chlorophenols, herbicides and priority pollutants. She developed and implemented clinical screening tests for volatile organics in biological samples and was experienced in the development and preparation of grant proposals funded by government and private agencies. Ms. Antoine has had numerous publications, presentations and technical reports over the course of her career.

FRANKLYN F. PIEHL, JR.

POSITION	Manager, Gas Chromatography and Organic Prep departments, ETC/Gulf South, New Orleans, LA.
EDUCATION	B. S., Chemistry, University of Michigan, 1980.
PROFESSIONAL AFFILIATIONS	Louisiana Water Pollution Control Association
PROFESSIONAL EXPERIENCE	Manager, Gas Chromatography Department, ETC/Gulf South, New Orleans, LA 1989 - present.
	Environmental Laboratory Manager, SGS Control Services, Inc., New Orleans, LA 1987 - 1989.
	Staff Chemist, SGS Control Services, Inc., New Orleans, LA 1985 - 1987.
	Staff Chemist, IT Analytical Services, Baton Rouge, LA 1984 - 1985.
	Chemist, Shilstone Testing Laboratory, New Orleans, LA 1980 - 1984.

EXPERIENCE SUMMARY

As Manager of the Gas Chromatography Department at ETC/Gulf South, Mr. Piehl manages the operation and maintenance of the GC instrumentation. As Organic Prep Manager, he is responsible for meeting hold times for samples and seeing samples are prepared according to prescribed procedures. He prioritizes the workload and makes work assignments for the laboratory analysts. He is responsible for data review and problem solving. He also is responsible for recruiting and supervises the training of chromatography and sample preparation laboratory personnel. In addition to GC, GC/MS and Organic Prep operations, Mr. Piehl has a broad spectrum of experience in the environmental laboratory including water quality, metals analysis, and computer application.

Mr. Piehl also serves as Technical Project Manager (TPM) for commercial projects that involve GC expertise. As TPM, he is responsible for resolving technical issues which arise, such as quality control excursions or changes in project scopes of work, and answers clients' technical questions.

At SGS Control Services, Mr. Piehl served as supervisor for the Environmental Laboratory. While there he installed analytical protocols for water, soils/sludges and hazardous wastes; selected, purchased and installed new analytical instrumentation; designed and installed the Quality Assurance program; and designed the reporting formats and procedures. He also served as the client contact for environmental projects.

At IT Analytical Services, Mr. Piehl was a staff chemist in the Baton Rouge field laboratory. He operated the Finnigan GC/MS and chromatography instrumentation performing volatile organics analyses. He also participated in field sampling activities.

KEITH RHODE

POSITION	Department Manager, Inorganics Laboratory, ETC/Gulf South, New Orleans, LA.
EDUCATION	Master of Science, Analytical Chemistry, University of New Orleans, 1983.
	Bachelor of Science, Chemistry, University of New Orleans, 1976.
PROFESSIONAL EXPERIENCE	Inorganics Manager, Inorganics Laboratory ETC/Gulf South, New Orleans, LA, 1992 - present.
	Manager of the General Chemistry Laboratory, EIRA, 1991 - 1992.
	Senior Chemist, Metals Laboratory, General Engineering Laboratories, Inc., 1990 - 1991.
	Manager, Inorganics Laboratory, Gulf South Environmental Laboratory, New Orleans, LA, 1986 - 1990.
	Research Associate for the University of New Orleans for the Center for Bio-Organic Studies, Metals Task Leader, New Orleans, LA, 1983 - 1986.
	Instructor, University of New Orleans, LA, 1980 - 1983.
	Graduate Assistant, University of New Orleans, LA, 1976 - 1980.
	Research Assistant, University of New Orleans, LA 1973 - 1975.

EXPERIENCE SUMMARY

As Manager of the Inorganics Department, Mr. Rhode is responsible for ensuring that all activities in the department meet safety, quality and production requirements. He manages all department procedures and processes to ensure completion of all assignments and tasks on schedule and in compliance with established Quality requirements. Mr. Rhode is also responsible for maintaining SOPs for all processes in the Inorganics department.

While working at EIRA as the Manager of the General Chemistry Laboratory, Mr. Rhode was responsible for maintaining the highest quality, efficiency, and effectiveness of the laboratory through strict compliance to methodologies in accordance with EIRA SOPs and QAPP. His responsibilities also include staffing, facilities, and budgeting within the Inorganics Laboratories. He was also responsible for troubleshooting, upgrading, and developing analysis techniques.

At General Engineering Laboratories, Mr. Rhode worked as a Senior Chemist in their Metals Lab. He was responsible for methods development and troubleshooting for ICP, ICP-MS, and AA with emphasis on environmental applications.

From 1986 to 1990 Mr. Rhode was employed by Gulf South Environmental Laboratory, Inc. as the Inorganics Manager. He was responsible for all aspects of the Inorganics department from scheduling of work to data review and technical reporting. Extensive knowledge of EPA CLP Inorganic Protocols, RCRA and NPDES regulations was obtained, as well as experience in AA, Graphite Furnace AA, DCP, ICP and CIP-MS.

As a Research Associate for the University of New Orleans, Mr. Rhode was responsible for the preparation and analyses of environmental and biological samples for trace metals by atomic absorption spectroscopy. He has had extensive experience with gas chromatography.

While still attending school, Mr. Rhode held various positions at the University of New Orleans in the Chemistry Department. He has gained practical experience working as a Research Assistant, Graduate Assistant, and Instructor.

DARRYL F. MELANCON

POSITION Systems Administrator, Systems Department, ETC/Gulf South, New Orleans, LA.

EDUCATION University of New Orleans, Bachelor of Science, Computer Science Candidate.

PROFESSIONAL EXPERIENCE

Systems Administrator, Systems Department, ETC/Gulf South, New Orleans, LA, 1990 - present.

Systems Administrator, Systems Department, Gulf South Environmental Laboratory, Inc., 1988 - 1990.

Systems Administrator, Analytical Chemistry Department, Gulf South Research Institute, New Orleans, LA 1987 - 1988.

Meterological Technician, United States Navy, 1976 - 1980.

EXPERIENCE SUMMARY

As the Systems Administrator, Mr. Melancon is responsible for the installation, implementation and modification of several different software packages wuch as: UNIX, ORACLE, Formaster, Telecations, WordPerfect, Lotus1-2-3, Harvard and FreeLance Graphics.

While in the U.S. Navy as a Meteorological Technician, he was an operator of the National Environmental Data Systems (NEDS) and telecommunication equipment. Also his responsibilities entailed hourly weather observation and flight pilot weather reporting.

FAYE CLESI

POSITION	Accountant, Accounting Department, ETC/Gulf South, New Orleans, LA
EDUCATION	Louisiana State University, B.S. Accounting, 1984
PROFESSIONAL EXPERIENCE	Accountant, Accounting Department, ETC/Gulf South, New Orleans, LA, 1988-present.
	Accounting Supervisor, American Bank and Trust Company, New Orleans, LA, 1987-1988.
	Investment Accountant, American Bank and Trust Company, New Orleans, LA, 1987.
	Accountant, American Bank and Trust Company, New Orleans, LA, 1986-1987.

EXPERIENCE SUMMARY

Ms. Clesi is employed by ETC/Gulf South, as an Accountant in the Accounting Department. She is responsible for processing purchase orders, accounts payable and accounts receivable. Additional duties consist of preparing daily and weekly invoice/login reports for the General Manager.

At American Bank and Trust Company, Ms. Clesi held various positions that assisted her in becoming Accounting Supervisor for the company. She was responsible for supervising accounts and completing various projects such as cash flow projections. Ms. Clesi also assisted the Vice President of Finance in developing and implementing a micro-computer based cost accounting program to allocate overhead costs to all revenue centers.

LABORATORY QUALITY ASSURANCE PLAN

Prepared by PACE Incorporated
New Jersey Laboratory (PACE/NJ)
284 Raritan Center Parkway, P.O. Box 7808
Edison, New Jersey 08818-7808
(908) 225-6700

PACE/NJ Approval

David M. Speid — 19 Sep 94
General Manager — Date

Stephen R. Grant — 9/14/94
Operations Manager — Date

[signature] — 9/14/94
Quality Assurance Manager — Date

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Document No.

This document has been prepared for the PACE Incorporated facility in Edison, New Jersey. The material contained herein is not to be disclosed to, or made available to any person or persons without prior written approval by authorized PACE/NJ personnel.

FOREWORD

The PACE Incorporated (PACE) laboratory in New Jersey (formerly ETC/Edison) has provided high quality analytical and data management services to major industrial corporations, waste disposal firms and governmental agencies since 1981. The laboratory's specialized services are directed at the accurate measurement of contaminants in water, soils and sediments, and hazardous wastes. Laboratory services have been developed in direct response to regulations written and enforced by federal and state environmental agencies. The laboratory has historically performed special project investigations that have enhanced its competence and versatility in the field of analytical chemistry.

PACE's New Jersey Laboratory (PACE/NJ) is part of the new PACE system of environmental testing facilities. The system, currently consisting of eighteen regional laboratories nationwide, has extensive analytical capacity, capability and expertise. The PACE headquarters is located in Minneapolis, Minnesota. Figure 1.1 includes all current PACE locations. This written quality assurance plan documents the procedures used to manage custody elements and analytical processes at PACE/NJ, and is consistent with the level of quality and integrity represented by the PACE organization.

This quality assurance plan has been prepared to conform in content with the USEPA QAMS-005/80, "Interim Guidelines and Specifications for Preparing Quality Assurance Project Plans", December 29, 1980. QAMS-005/80 includes a section titled *Project Description*. In this document, the laboratory policy statement replaces specific project description information. The tables and figures referenced throughout the document are found in the appendix. The appendices of project specific quality assurance documents may include additional elements or information.

This document describes ongoing laboratory operations for routine analyses performed at PACE/NJ. As such, the material contained within is subject to change. Changes may be based on specific project requirements or procedural system modifications geared towards operational process and quality improvements. This document is reviewed and updated on a minimum yearly basis.

Because of the variability of samples and matrices, it is not unusual to find that the performance criteria of a particular method is unachievable on particular samples. In such circumstances, any method or criteria modification will be noted in each final report.

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APPENDIX

Figures & Tables

The "ETC" name may appear on PACE/NJ forms. Form revisions are in process.

STATEMENT OF POLICY

PACE/NJ is committed to meeting the quality standards of the regulatory environmental laboratory industry and the regulated community. PACE/NJ's objective is to consistently produce analytical data which is of known quality and meets the quality objectives of the methods and the data user. This will enable the data user to make rational, confident, cost effective decisions on the assessment and resolution of environmental problems.

Laboratory Ethics

PACE/NJ has a written ethical code that all employees are required to adhere to. The code describes the high scientific and personal standards necessary to ethically conduct business in the industry. PACE/NJ policy requires that all employees sign an ethical conduct agreement (Figure 3.1), binding them to the principals of the code. The laboratory's Code of Ethics is presented below.

Code of Ethics

PACE/NJ provides analytical chemistry services on environmental matters to the regulated community. The data the company produces provides the foundation for determining the risk presented by a chemical pollutant to human health and the environment. The environmental laboratory business is dependent upon the accurate portrayal of environmental chemistry data. The process is reliant upon a high level of scientific and personal ethics. Accordingly, PACE/NJ has adapted the following ethical code to which each employee is expected to adhere.

- To search for scientific truths by use of the scientific method.
- To be faithful and incorruptible, respecting confidence, advising honesty.
- To maintain professional integrity as an individual.
- To place the public welfare above any considerations of self-interest recognizing and responding to community concerns.
- To present services in a confidential, honest, and forthright manner.
- To produce results that are accurate and defensible.
- To comply with all pertinent federal, state and local laws and regulations as it relates to his or her practice.
- To provide employees with guidelines and an understanding of the ethical and quality standards required to work in this industry.

ORGANIZATION AND RESPONSIBILITY

The PACE/NJ organizational structure (Figure 4.1) allows close, coordinated interactions and promotes the common goals of successful project management and quality data. PACE/NJ's quality assurance group is independent of laboratory operations. Each individual in the PACE/NJ organization is responsible for their product or the service they perform. Analysts and technicians who handle samples or analytical data have the following minimum responsibilities.

- Performs the expected services and methodologies.
- Performs the quality requirements of their tasks.
- Takes the corrective actions described in the analytical methods or protocols when the quality control specifications are not met.
- Accurately communicates any sample or quality problems to responsible management personnel.
- Ensures that sample custody is maintained.
- Adheres to the PACE/NJ Code of Ethics.

Project Organization

Projects at PACE/NJ are monitored by project teams selected prior to project initiation. The teams consist of representatives from the laboratory operations, quality assurance and marketing staff. The goal is to set up and monitor the project to meet client needs. Figure 4.2 presents the key individuals on the PACE/NJ project team. The minimum responsibilities for key project personnel are as follows.

Account Executive

- Supports client regulatory programs.
- Coodinates pre-project meetings.
- Establishes contractual terms and conditions.
- Provides oversite of the project.
- Communicates the client's quality needs.

Project Manager

- Serves as primary client contact for the project.
- Provides advisory consultation to clients on regulatory and technical issues.

- Attends pre-project and project progress meetings; assists in client or project audits.
- Defines project scope through detailed documentation of client and project quality and technical requirements.
- Communicates the client and project quality and technical requirements to laboratory personnel; implements the project.
- Coordinates field activities with sample management personnel to ensure proper delivery of shipping containers, sample bottles, etc.
- Tracks and manages the project through the laboratory.
- Updates the client on nonconformances and responds to requests for information.
- Coordinates technical report generation to ensure client commitments are achieved.

Operations Manager

- Provides supervision of laboratory operations.
- Implements the laboratory quality assurance plan.
- Ensures proper scheduling and execution of testing programs.
- Assures that quality assurance and quality control criteria of analytical methods and projects are satisfied.
- Assesses data quality and takes corrective action when necessary.
- Notifies the project team of specific laboratory nonconformances and changes.
- Approves and releases technical and data management reports.
- Ensures that analysts and technicians maintain sample custody in the laboratory.
- Approves project specific laboratory quality assurance plans.
- Coordinates management of projects through technical supervisors.

Quality Assurance Officer:

- Serves as the official organizational contact for quality assurance matters. Concerns may include, but are not limited to, quality assurance plans, standard operating procedures, analytical methodologies and protocols, audits, certifications, support services, and corrective actions.
- Identifies and responds to quality assurance needs, assists in problem resolution and answers requests for guidance, information or assistance.

- Provides guidance in the development of quality assurance plans.
- Reviews, evaluates and approves written quality assurance plans.
- Tracks the progress of quality assurance tasks (from preplanning to data assessment) and consults periodically with project managers.
- Monitors quality assurance/quality control functions and corrective actions throughout the laboratory facility.
- Provides the General Manager reports of laboratory performance.

QUALITY ASSURANCE OBJECTIVES

The PACE/NJ laboratory's quality assurance program has two primary aims.

- Ensure that the laboratory produces data which meets the quality requirements of the methods in use and the quality objectives of the data user. By accomplishing this objective, the data user will be able to employ the laboratory data for its intended use.

- Provide management with data quality and operational performance feedback. Performance feedback data enables management to determine if the laboratory facility is achieving the established quality and operational standards of the environmental laboratory industry. It enables PACE/NJ to assess operational performance from a quality perspective and perform corrective action as necessary.

The quality assurance objectives of the laboratory must be consistent with specifications for analytical services cited for projects and samples. The laboratory will perform analytical services and support in accordance with project requirements as specified by the client.

The data quality objectives of the laboratory are to produce complete, valid, and verifiable data. PACE/NJ's goals are to execute the required methodologies and procedures, and to generate precise and accurate measurements. Tables 5.1, 5.2 and 5.3 identify the laboratory's routine quality control objectives.

SAMPLING SERVICES

Field Support

PACE/NJ provides shipping containers (PACE/NJ Sample Shuttle™), custody documents, custody seals, sample bottles, labels, chemical preservatives for water samples, "blue ice" packs to maintain thermal preservation, and trip and field blanks to support field sampling events. Table 6.1 lists sample container types, preservatives and holding times. PACE/NJ can provide shuttle pick up and delivery services to its clients.

Upon receipt of the field samples at the laboratory, PACE/NJ ensures that sample bottles are maintained according to preservation requirements and that sample storage conditions do not contribute to the presence of test analytes in the samples.

PACE/NJ Shipping Containers

The PACE/NJ Sample Shuttle™ was developed in 1981 by PACE/NJ staff for the transport of environmental samples from the field to the laboratory. The Sample Shuttle is a rugged carrying case lined with insulating polyurethane. Insulating sleeves with pre-formed slots hold the sample bottles. The container is lockable from the outside. Chain-of-custody seals and forms, employed for each Shuttle packed at PACE/NJ, ensure complete documentation and provide evidence of unbroken custody of the Shuttle contents. The Shuttle meets or exceeds all protocol requirements (i.e., DOT, USEPA, ASTM) for shipping. Figure 6.1 lists instructions for Sample Shuttle use and sampling notes. The Shuttle is configured at the laboratory to provide the client with all of the sample containers needed for the analyses.

PACE/NJ Shuttles and/or commercial coolers are utilized for projects based upon the preference of the client. The commercial coolers are, likewise, sealed and provide the above listed items for sampling events and custody documentation. In this document, generic references to Sample Shuttles will apply to commercial coolers as well.

Preservation

PACE/NJ provides the required chemical preservatives for water samples and "blue ice" packs, for thermal preservation at 4 +/- 2° C, in the shipping containers during the shipping process. High quality reagent grade chemical preservatives are used. The ice packs are supplied at pre-frozen or ambient temperatures based upon the client's needs. It is the responsibility of those collecting the samples to properly use these materials and ensure that proper preservation techniques are performed and preservative requirements are met. PACE/NJ recommends that all sample containers be chilled with ice after collection prior to shipment in the Sample Shuttles.

Upon receipt of samples at the laboratory, the temperature of each Shuttle is measured and recorded on the chain of custody documents. Similarly, the pH of bottles to which chemical preservative was added is measured (with the exception of sample collected for volatile organic compounds), and the check recorded. A disposable pipette is used to remove an aliquot of the sample for the pH check. When deviations from the required chemical or thermal preservation are noted, the Technical Project Manager is notified, and clients may become involved with determining a course of action to follow.

Water samples for GC and GC/MS volatile aromatics determinations are monitored for pH within 24 hours of sample log-in. The pH is initially checked by testing the contents of the sample bottles used during volatiles screening procedures. The remaining bottles are not opened until analysis, at which time the pH of each individual sample bottle used is checked. Samples that have an observed pH > 2 upon initial monitoring are scheduled for analysis with a shortened hold time of 7 days from sampling. The portion of sample used for the analytical determination is removed from the vial prior to checking the sample pH. Sample pH measurements are recorded on laboratory chronicles as they are taken.

Sample Bottles

PACE/NJ provides pre-cleaned sample bottles in the shipping containers for sample collection. Used sample bottles are never reused by the laboratory. Vendor prepared 200™, 300™ or equivalent bottles can be provided as projects necessitate. Laboratory cleaning and preparation procedures for commonly used bottles are as follows.

Bottle Caps: Teflon lined. New caps are rinsed with deionized water, allowed to air dry in racks, then placed on bottles.

Amber Glass Bottles (1 L, 500 ml, 250 ml): Rinsed with de-ionized water. Baked at 200° C for 30 minutes prior to capping and use.

Clear Glass Vials (40 ml for volatile organics): Teflon lined septa for caps are stored in containers prior to use. Bottles, caps and septas are rinsed with de-ionized water, then baked for one hour at 105° C in an oven used exclusively for this purpose. Hydrochloric acid preservative is added to each vial used for aqueous samples. Upon client request 20 mg of sodium thiosulfate is added to remove residual chlorine in the sample. Bottles are capped and stored in sealed, air-tight metal containers prior to use. Bags of granulated carbon are enclosed to adsorb any organic vapors present.

Amber Glass Bottles (125 ml): Rinsed with de-ionized water and air dried. Baked at 200° C for 30 minutes prior to capping and use.

Plastic Bottles: Bottles and caps are rinsed with deionized water and allowed to air dry in racks prior to capping and use. Bottles used to collect sample for the analysis of metals are rinsed in nitric acid solution.

Coliform Bottles: All bottles used for the sampling and analysis of coliform are purchased sterilized. They are received sealed and autoclaved.

Sample Receipt Schedule

Samples are normally delivered to the PACE/NJ facility during normal business hours within one day following field sampling unless different arrangements are made in advance with an authorized PACE/NJ representative. Shipping containers received at the laboratory on business holidays, weekends or after normal work hours will be placed in the walk-in refrigerator and opened on the next regular business day unless prior arrangements are made in advance for day's receipt and log-in.

SAMPLE CUSTODY

Areas of Concern

The Chain of Custody in the laboratory consists of two areas of concern:

- Receipt and Log-in of Samples; and
- Maintaining Internal Custody of samples transferred throughout the laboratory.

Samples are physical evidence and, as such, are handled at PACE/NJ according to certain procedural safeguards. For some types of legal proceedings, a showing to the court that the custodial laboratory is a secure area may be all that is required for sample data to be admitted as evidence. In other cases, the court may require a detailed showing of the hand-to-hand changes of custody that a sample has undergone. Federal and state agencies and private sector clients may also require varying levels of custody documentation from the laboratory. PACE/NJ is equipped to provide the defined level of custody documentation necessary.

Maintaining Custody by NEIC Definition

Custody is maintained by USEPA National Enforcement Investigations Center (NEIC) definition when:

- The sample is in the actual possession of the responsible person, or
- The sample is in the responsible person's view after being in their possession, or
- The sample is in the responsible person's possession and then they locked or sealed it up to prevent tampering, or
- The sample is in a secure area.

PACE/NJ Laboratory Procedures

To satisfy these [illegible] provisions, the following standard operating procedures are employed:

- The PA[illegible]tory is maintained as a limited access, secured facility.
- The grounds and parking areas around the building are patrolled on a regular basis by a roving patrol supplied by the Raritan Industrial Center.
- A security guard is on duty inside the building evenings on Monday through Friday. After his departure through 7:00 am (Monday through Friday), and on weekends and holidays, the building is secured and its perimeter is under electronic surveillance by a local security service.
- Only PACE/NJ managers are authorized and have keys to open the building during off-hours when the guard is not on duty.

- Employee access to the building and selected security area (including walk-in refrigerators) within the building is controlled by a computerized card reader employee identification system. Access is on a need basis during authorized work hours.

- Visitors must register upon entering the lobby of the facility, and must be accompanied by their host at all times while they are in the building.

- Receipt and log-in of samples is fully documented and is performed in a controlled area.

- Samples are stored in a secure area.

- Walk-in refrigerators, freezers, and other primary sample storage areas are locked at all times or when unattended, dependent upon the function of the unit.

- Only designated PACE/NJ personnel have access to the primary sample storage areas.

- Samples remain in secured sample storage until removed for sample preparation or analysis.

- The internal transfer of samples is controlled and documented.

PACE/NJ Sample Custodians

PACE/NJ Sample Custodians are responsible to perform the following:

- Receive, inspect and record the condition of samples and shipping containers.

- Sign appropriate documents shipped with the samples.

- Verify and record correctness of sample documentation (for example, chain of custody: seal is intact, chemical preservatives are added, etcetera).

- Initiate transfer of samples to appropriate lab sections with proper documentation (for example, loglink, special instruction sheets, laboratory identification numbers, etcetera).

- Place samples in appropriate storage and secure areas.

- Control and monitor access and storage of samples.

Custody Using the PACE/NJ Sample Shuttle™ System

The PACE/NJ Sample Shuttle™ system establishes and maintains the integrity of contents from the time that the Shuttle is packed and shipped from PACE/NJ through shipment, sampling, transport, and return to PACE/NJ via its enclosed paperwork and seal system (refer to Section 6.0).

The laboratory initiates the custody procedure and assigns unique PACE/NJ sample identification numbers to the client samples. The PACE/NJ sample numbers are utilized during laboratory shipping, receipt and log-in procedures. Coded descriptions of the required analytical and support

services are associated with each sample number. Upon receipt each sample bottle is labelled with the PACE/NJ sample number, the analysis type, the site location/facility code and chemical preservative, as applicable. The PACE/NJ sample number is recorded on the chain of custody and log-in forms and is used to track the sample pathway throughout the analytical process.

During log-in, the Sample Custodian checks the contents of the shipping containers against the PACE/NJ custody documents, Chain of Custody Form 1 (CC1) for single samples or Chain-of-Custody Chronicle for multiple samples (Figure 7.1 - 7.3), or other client supplied custody records. Observations concerning the presence or absence of bottles or the condition of the samples, as received, are recorded on the custody forms. The original custody form is signed by the Sample Custodian, documenting sample receipt by the laboratory. Completed custody forms are placed into file folders identified by unique log-in codes specific for the samples. The file folders are transferred to report production personnel who incorporate the custody forms into the report packages, and assume responsibility for proper PACE/NJ custody records archive along with the sample data.

Sample Receipt and Log-In

The PACE/NJ employee who accepts receipt of client samples upon arrival at the laboratory is a designated Sample Custodian. The Sample Custodian examines both the shipping containers and the Chain of Custody and shipping documents. The Sample Custodian is responsible for the receipt and log-in operations. The custody seal and documents are examined for compliance. Any and all noncompliances are documented and the client contacted.

Should the samples arrive before all the necessary information is received from the client regarding analysis, the clock will not start (days will not be counted towards turnaround) until the information is obtained; actual sample holding time is independent and not affected by any delay. Turnaround is defined as the time interval between laboratory receipt of a sample in a condition suitable for the prescribed analysis and delivery of analytical results to the client.

Sample turnaround may be based upon single sample receipt at the laboratory or the receipt date of the last sample of a sample delivery group (SDG) received at the PACE/NJ facility. SDG is defined by the following, whichever is more frequent: each twenty (20) field samples received or each fourteen (14) day calendar period during which field samples are received following the first in the group. Samples may be assigned to SDGs by matrix (aqueous and soils/sediments placed in separate SDGs) at the discretion of the laboratory. The turnaround time is based upon the analytical protocol or as defined by the client.

The PACE/NJ Shuttle or other shipping container is opened, the temperature taken, and all sample containers checked against the accompanying paperwork. The pH of sample aliquots which have been chemically preserved, except those used in volatile organics analyses, are monitored. The client is contacted by telephone regarding any custody problems or problems with the condition of the samples upon receipt at the laboratory. Receipt documentation is completed and an PACE/NJ sample log-in form (Figure 7.4) is generated. A log-in form reflects the information present in the laboratory computer system for the sample.

The log-in form is an internal document with a unique index number designed to summarize all the relevant information concerning a sample's receipt and analytical requirements. It is

circulated internally and instructs the laboratory with regards to the receipt, and the required analyses and reporting of the sample. The index number is referred to as the log-in or log-link number. Each sample bottle is labeled with its log-in number reference.

After the samples are logged-in, the sample bottles are placed in designated areas of the cold storage units. The sample bottles are stored according to preservative type and analyses in log-link order. Volatile organic sample bottles are stored in a separate refrigerator unit.

Laboratory Custody for Client Drop Off of Samples

Transfer of custody of the shipping containers is documented by signature of the sample custodian and the client or their designate (Figure 7.5). If the client desires, the shipping containers are opened and the samples are itemized, making all of the same notations as stated above.

The client is then given the completed sample Chain of Custody to review and approve the recorded information. The client signs the Chain of Custody, relinquishing the samples to the Sample Custodian. A copy of the Chain of Custody serves as a receipt for the client if one is requested. The standard log-in procedures previously described follow.

Laboratory Custody for Cooler/Box by Carrier

The carrier and the time of arrival at PACE/NJ is recorded on the airbill. The number of items on the airbill is checked with the actual number received to make sure all shipping containers arrived. An PACE/NJ Chain of Custody Form is created if not already provided (Figure 7.6). Notation is made as to whether the container is sealed and if there are any specific types of seal involved.

The container is then opened, the temperature taken and the contents (samples) are itemized. The following information is recorded on a Chain of Custody Form: sample point identifications, sample time, the condition of the samples, the volumes received and the preservation. The completed custody document is signed, demonstrating acceptance of custody by the PACE/NJ Sample Custodian. If there is any type of custody document enclosed with the samples, this chain of custody is also completed, noting the above information. The standard log-in procedures previously described follow.

Special Handling for High Hazard Samples

If a shipping container is labeled "Caution - Hazardous Materials," or if it contains samples to be analyzed for extremely hazardous materials, the department supervisor is notified prior to log-in. The containers are opened by Sample Custodians who have successfully completed respirator fit-test and safety training sessions in the necessary protective equipment. The shipping containers are opened using all the safety measures deemed necessary by the PACE/NJ Safety Committee and department supervisor.

PACE/NJ sample receipt and log-in procedures are closely followed. The receipt of the sealed containers are documented on the appropriate chain of custody form. The persons authorized to open such containers will perform the log-in steps previously described in this section.

Subcontract Laboratory Chain of Custody

Samples are subcontracted by PACE/NJ to approved laboratories for client required analyses that, for example, the PACE/NJ laboratory does not regularly perform. Subcontract laboratory custody is documented on the PACE/NJ Subcontract Chain of Custody form (Figure 7.7). Samples are shipped by PACE/NJ using overnight carrier services, picked up daily by the subcontract laboratory or delivered by PACE/NJ, depending on the subcontract laboratory utilized for the analyses.

Maintaining Internal Chain of Custody

Sample custody within the PACE/NJ facility is documented on laboratory records by the sample custodians and the authorized PACE/NJ personnel who take custody of the samples to perform the required preparation and analytical procedures. A number of internal custody records may exist for a sample for which multiple determinations are performed. Internal custody is established by the PACE/NJ Sample Custodian.

Changes of custody within the laboratory are recorded on the Internal Custody Form (Figure 7.8) as they occur. The date and time that sample custody is assumed and relinquished is documented. Personnel signatures authenticate the accuracy of the information recorded on the form. The Appendix additionally includes PACE/NJ Laboratory Chronicles for organic and inorganic preparations and analyses. The laboratory staff uses these documents (Figures 7.9 through 7.15), which offer further demonstration of internal custody while the samples are being worked on in the laboratory.

Sample Tracking

The following procedures are used to track samples. Both the preparation and the analysis of samples are documented through the use of Laboratory Chronicles and Internal Custody Forms as previously described. Computer reports are generated daily showing the status of each sample for all analyses as updated by the responsible scheduler. Sample status information is accessed electronically through the PACE/NJ computer data base as well.

When an analytical batch is completed, the transfer of the sample results from the analytical area to the technical report production area is recorded in a log notebook. When the technical reports are complete, a tracking system is utilized to mail the reports and archive them according to procedures identified in Section 10.

Sample Custody After Analysis

The laboratory custody of a sample routinely ends with laboratory disposal. PACE/NJ's routine disposal of samples occurs after a minimum of thirty days from the mail date of the sample technical report(s). PACE/NJ retains samples for longer periods of time to comply with client or contract requirements, and returns remaining sample to clients at their request. At PACE/NJ's discretion, hazardous samples may be returned to clients. Sample disposal is addressed at greater length in Section 9.0, Waste Disposal.

CALIBRATION PROCEDURES AND FREQUENCY

Instrument calibration is a mandatory requirement of performing quantitative analytical methodology. The laboratory must meet the established method criteria for instrument calibration and calibration verification before proceeding with sample analysis. Table 8.1 summarizes the laboratory's routine instrument calibration procedures.

PACE/NJ will meet the calibration criteria specified in the methods. The analysts will not continue with an analysis or accept data unless the calibration requirements have been met. Likewise, when an analytical method or protocol includes the use of other instrument quality control procedures, such as GC/MS tuning, the analysis will not proceed unless the quality control criteria are met.

Analytical Reference Standards

PACE/NJ's analytical reference standards are foundational to the quality of the analytical determinations performed. Instrument calibration and calibration verification is performed at the method required frequency utilizing analytical reference standards that satisfy the method or protocol specifications. Laboratory pure water and reagent grade or higher organic solvents and acids are used for solutions. Proper storage and handling techniques are followed. Standards are not used past their expiration dates.

Organic standards are obtained from a variety of vendors. Stock solutions or working calibration standards are prepared from purchased neat materials or concentrated solutions. Several custom working standards are purchased with the components at the desired concentrations. PACE/NJ lot number designations can be used to trace reference standards to their purchased sources. PACE/NJ's written documentation provides in-house traceability. Percent purity traceable to NIST and USEPA may be available from vendors. Vendor prepared standards utilized for several analytical protocol are purchased as certified by USEPA Contract Laboratory Program (CLP) procedures and criteria for analytical reference solutions.

Inorganic standards are obtained from vendors that specify traceability to NIST and USEPA materials. Most are purchased as solutions and diluted at the laboratory. PACE/NJ's written documentation provides in-house traceability of the working reference solutions to their purchased sources. Lot numbers are used for several applications.

Both organic and inorganic standards preparations are thoroughly documented in laboratory notebooks or on pre-printed log sheets designated for that purpose. Preparation and expiration dates are indicated. The preparer signs, authenticating the laboratory entry. Recorded information includes: the concentrated source; the volume or weight of the source used in the dilution; the final volume and concentration level of the dilution; the acid, preservative or organic solvents used, etcetera. Dilution factors are recorded for several applications. Cross reference to other sources of information may be included in the documentation scheme. The preparation documentation is retained by the laboratory should it be needed for verification at a later date.

ANALYTICAL PROCEDURES

Methodologies

The PACE/NJ laboratory utilizes approved USEPA methodologies for all analyses, if available and applicable. The deliverables included in the sample technical reports are based upon the level of report deliverable requested and the quality assurance requirements of the methods performed. Analytical results and quality assurance protocols are based upon, but not limited to, the following methods and guidelines.

"Methods of Organic Chemical Analysis of Municipal and Industrial Wastewater", Federal Register Vol. 49, No. 209, October 26, 1984;

"Test Methods for Evaluating Solid Waste, Physical/Chemical Methods", SW-846 Third Edition, Revision 0, USEPA, September 1986;

"Standard Methods for the Examination of Water and Wastewater", 17th & 18th Editions, American Public Health Assoc., American Water Works Assoc., Water Pollution Control Federation, 1989 and 1992;

"Methods for Chemical Analysis of Water and Wastes", EPA 600/4-79-020, EMSL, March 1983;

"Methods for the Determination of Organic Compounds in Drinking Water", EPA-600/4-88/039, EMSL, December 1988 with July 1991 revisions;

USEPA Contract Laboratory Program Statements of Work for Organics Analysis: SOW OLM01.0 and latest published revisions, 1990; SOW February 1988;

USEPA Contract Laboratory Program Statements of Work for Inorganic Analysis: SOW ILM02.1, September 1991; SOW ILM01.0 and latest published revisions, 1990; SOW July 1988;

"Handbook for Analytical Quality Control in Water and Wastewater Laboratories", EPA-600/4-79-019, March 1979;

National Enforcement Investigation Center Policies and Procedures Manual, EPA-330/9/78/001-R, Revised May 1986; and

"Emergency Standard Practice for Generation of Environmental Data Related to Waste Management Activities", ASTM ES 16-90, American Society for Testing and Materials, June 1990.

PACE/NJ may revise procedures to reflect more recent method revisions or USEPA CLP Statements of Work (SOW) prior to or during a project, and reserves the right to utilize later revisions or SOW for project samples at that time.

In addition to performing standard methods and procedures, PACE/NJ has the capability of modifying methods, as well as performing specific client or project methods. Within normal laboratory operations, PACE/NJ can design experiments or method improvements to offer its clients improved performance for a specified parameter.

Laboratory Glassware

The laboratory utilizes accepted procedures for cleaning laboratory glassware. Cleaning procedures differ based on the intended use of the glassware but always include washing in laboratory grade phosphate-free detergents, tap water rinses followed by deionized or other laboratory grade water rinse, and acid or solvent rinses or soaks as appropriate for the glassware's intended use.

After use, laboratory glassware is rinsed to remove contaminants prior to being placed in the glassware area for cleaning. Glassware is manually or machine washed according to specifications; all signs of visible discoloration and/or any materials that may have been present must be eliminated or the glassware is discarded. Glassware may be baked at a high temperature (400° C) to remove any potential residuals, depending on the piece and its intended use. Glassware is properly stored prior to use to ensure that contamination does not occur, and is solvent or acid rinsed as appropriate prior to use.

Materials Procurement and Control

Purchasing guidelines for all materials and performance guidelines for equipment having an effect on data quality are established. Materials are purchased according to method specifications or other regulatory or contract criteria; purchase order substitutions are not permitted by the purchasing department without prior expressed approval by authorized operations management.

Goods and materials are visually inspected upon receipt to ensure they are the items that were ordered from the vendor. Discrepancies are checked; materials that do not satisfy purity, quality or grade requirements are returned. Reagent lot number information are recorded on laboratory log sheets; clean method blanks indicate that reagents utilized for an analytical determination did not contribute to the presence of an analyte in a sample.

Chemicals and reagents are stored in accordance with applicable fire and safety regulations and guidelines, protocol requirements and under conditions specific to the material or its use. Areas dedicated for chemical reagent storage are well maintained and orderly; materials maintained by bench personnel are expected to be inventoried and stored in a similar manner.

The laboratory realizes the importance of proper storage and documentation procedures for both analytical reference standards and chemical reagents. Chemicals and reagents arriving at the facilities in vendor shipments are dated upon receipt to establish their order of use and minimize the possibility of exceeding their useful shelf life. Likewise, analysts and technicians are trained to date materials upon opening as a tracking mechanism for the material.

Periodically, inventories are reviewed and materials are evaluated for disposal. Materials that are received without a manufacturer's expiration date are assessed based upon receipt and opening dates and the nature of the material. When contamination is traced to a reagent, the bottle or entire lot is removed from service.

PACE/NJ Reagent Grade Water

PACE/NJ maintains several water purification systems within the laboratory that produce water of sufficient quality so as to be demonstrated acceptable for use in preparation of reagents and method blanks, and provided for sampling activities as field/trip/equipment/rinsate blanks. The same source waters that are used for ongoing laboratory analyses are provided for client use. Blanks that are provided to clients in sample shuttles must be prepared on the day that the shuttle is shipped from PACE/NJ. The water is monitored daily for specific conductance.

The PACE/NJ water purification system includes a reverse osmosis process incorporating carbon and deionizing tanks prior to the end use. Dependent upon the use, additional prefilters, carbon tanks, deionizing tanks and organic scavenger polishers may be used on the finished water. PACE/NJ water purification processes also include independent tank systems that are configured to generate a finished water suitable for its intended laboratory use.

Waste Disposal

PACE/NJ's waste disposal practices conform to all USEPA and NJDEPE waste handling requirements. The laboratory retains a RCRA Part A permit (generator only) which allows the laboratory to generate and store hazardous materials for a maximum of ninety days. Wastes are shipped to USEPA permitted and approved treatment, storage, and disposal facilities prior to expiration of the ninety day limit. Before engaging a waste disposal firm, PACE/NJ obtains copies of the firm's permits and all applicable identification numbers. The firm must also provide certificates of insurance to demonstrate adequate liability coverage. This information is updated on an annual basis.

Laboratory wastes are segregated into several waste streams. Aqueous wastes are treated as a single stream and removed to a waste treatment plant by a bulk handler. Solid material is segregated into soils/sludges and solid laboratory trash (not sample related) and removed to a subtitle C waste facility. PCB containing wastes (samples with known amounts or sample extracts containing PCBs) are removed to facilities permitted to accept TSCA wastes. Samples containing chlorinated dioxins are isolated and returned to the client. Alternatively, they can be disposed through an EPA approved disposal pathway. Oil/water mixtures and waste solvents are sent to a USEPA approved fuel blending facility. PACE/NJ maintains records of all waste transactions as well as all waste contractor documentation.

DATA REDUCTION, VALIDATION AND REPORTING

Data reduction, validation and reporting describes the processes that result in the delivery of quantitative analytical data to the data user. These processes include calculation of raw data into final concentration units, reviewing results for accuracy and assembly of the technical report contents for delivery to the data user. The following describes procedures employed at PACE/NJ for translating raw analytical data into accurate, finished sample reports and data storage.

Data Review and Processing Procedures

All organic and inorganic data generated by PACE/NJ are reviewed by designated trained personnel. The analysts who acquire the data are responsible for initial on-line checks for compliance to the analytical requirements. After a sample batch is acquired, the data review procedure includes data interpretation and quantitation, inspection of quality control data against criteria, data reduction, narrative or comments writing, and ensuring that the data package includes all required analytical and quality control results, raw data and laboratory chronicles. After review and acceptance, analytical results are entered into PACE/NJ's computerized data base and tabular summary tables are generated, or USEPA CLP forms are created.

The completed data package is transferred to a designated reviewer who performs a quality control audit for use of the proper methodology and detection limits, compliance to quality control protocol and criteria, presence and completeness of required deliverables, and accuracy of calculations and data quantitation.

Data packages are then transferred to the production service personnel who review each data package to ensure compliance with client orders by reviewing on-line input in the PACE/NJ computer tracking system. The laboratory data is assembled in the client's technical reports. Reports are reviewed for completion prior to reproduction in the copy/bind department. Figure 10.1 gives an overall view of the general operations flow.

Use of checklists ensure that all data is systematically handled and no steps are omitted. Checklists are reviewed, and are retained and accessible should they need to be referenced at a later date. The data and deliverables are checked and signed during processing procedures, and then systematically filed by reference identification numbers.

Technical Report Deliverables

Sample technical reports are prepared to include the components or level of deliverables requested by clients for samples or projects. PACE/NJ's standard report includes tabular results, data system printouts for several analyses, chain of custody records, and, as provided to the laboratory, sampling time and date, as well as field location code and client sample point identifications. Quality control results are routinely included for several analyses.

PACE/NJ also prepares technical reports that include full data deliverables for validation purposes, and lesser, abbreviated reports. Full deliverables include all raw and processed data applicable to the analyses performed. PACE/NJ prepares single sample technical reports or multi-sample report packages. The multi-sample technical reports contain results for a sample

delivery group (SDG) or other client or laborato: defined sample set. PACE/NJ recommends multi-sample reports when full deliverables packages are required.

Data Archive

Sufficient records are retained to recreate analytical events at the laboratory. Records are cataloged and maintained in limited access areas. Data archive and storage is managed by designated individuals who control the access to stored information.

All information retained at the PACE/NJ facility is stored in secured areas. All hard-copy information is stored on-site at the laboratory or off-site at a commercial document storage facility equipped with a professional security system. All electronic data is stored on-site at the laboratory or off-site at a commercial document storage facility equipped with a professional security system and a controlled environment suitable for storage of magnetic media.

PACE/NJ reserves the right to transfer hard-copy information onto microfilm. PACE/NJ reserves the right to store information in hard-copy files, on magnetic media and/or microfilm. The information is retained and accessible for a minimum of seven years unless otherwise specified through a client specific contract.

Response to Inquiries

The PACE/NJ laboratory recognizes the importance of its timely response to inquiries regarding the laboratory's work for samples and projects. The laboratory will respond to inquiries as rapidly as possible as part of its corrective action plan. PACE/NJ should be considered the primary contact for all data inquiries when subcontract or other network laboratories are used for analyses.

INTERNAL QUALITY CONTROL CHECKS AND FREQUENCY

The responsibility for the internal analytical quality control checks rests with the laboratory analyst. The type, frequency and acceptance criteria of the checks performed are based upon the reference analytical methodology employed and client or project requirements. In cases where the project required method does not address these critical issues, PACE/NJ may recommend that quality control protocols be established on a per method basis to meet the intended data quality objectives of the project.

The following descriptions present a summary of quality control samples that are used routinely for PACE/NJ projects. These data are compiled and are used both by the QA department and project staff to monitor data for systematic analytical problems.

Trip Blank: Analyte free reagent grade water prepared by the laboratory, shipped in the Sample Shuttle, and analyzed with the samples to detect accidental or incidental contamination during transport. Analyzed as required for a project. One trip blank is used per shipping container.

Field Blank: Reagent grade water provided by the laboratory that is transferred on-site to an additional clean sample container to evaluate the environmental or procedural effects of a sampling event; used to determine if contamination occurred during field sampling. Analyzed as required for a project.

Equipment Blank (Rinsate Blank): Reagent grade water provided by the laboratory that is passed through sampling equipment to determine the effectiveness of the field equipment cleaning procedures. Analyzed as required for a project.

Method Blank (Reagent Blank): A blank used to check chemical reagent or process introduced contamination in the laboratory. Analyzed, at minimum, at a 1:20 sample frequency or as required by a method if more often.

Quality Control Spiked Blank/Laboratory Control Sample: Secondary/independent source standard reference materials spiked into reagent grade water or other blank material and carried through the entire preparation and/or analytical process to verify or demonstrate method performance. Analyzed, at minimum, at a 1:20 sample frequency or as required by a method if more often.

Spiked Sample (Matrix Spike): A client sample spiked with standard reference materials and carried through the entire preparation and/or analytical process to evaluate sample matrix effects on analyte recovery and accuracy. Analyzed, at minimum, at a 1:20 sample frequency.

Unspiked Laboratory Duplicate: A client sample which is split and carried through the entire preparation and/or analytical process as a replicate sample to evaluate laboratory reproducibility and precision. Analyzed, at minimum, at a 1:20 sample frequency for metals and wet chemistry determinations.

Spiked Laboratory Duplicates (Matrix Spike & Matrix Spike Duplicate): A client sample which is split, spiked with standard reference materials, and carried through the entire preparation and/or analytical process as a replicate sample to evaluate sample matrix effects on analyte recovery and accuracy as well as laboratory reproducibility and precision. Analyzed, at minimum, at a 1:20 sample frequency for organics determinations.

The following are added to field and quality control samples for organic analyses:

Internal Standards: Compounds that possess similar chemical and physical properties to the target analytes. Added to samples or extracts prior to analysis, and used as retention and response reference points and to verify instrument performance. Evaluated as specified in the applicable GC/MS analytical methods. Used for several GC determinations.

Surrogates: Compounds that possess similar chemical and physical properties to the target analytes. Added to each sample to check for matrix effects or other difficulties related to method application. USEPA CLP and SW-846 recommended surrogates and recovery limits are used and reported when available.

PERFORMANCE AND SYSTEM AUDITS

Internal and external performance and system audits are used to assess the laboratory's ability to perform and support environmental analyses by evaluating it against required protocol or other stated objectives. Outlier values and investigative findings can result in corrective actions that are designed to cause improvement and prevent recurrences or lead to, for example, conformance to a specific future project's requirements.

A performance audit is a quantitative or qualitative evaluation of analytical data produced by a laboratory using samples containing analytes of interest. Performance audit samples, known also as proficiency or performance evaluation samples, are introduced to the laboratory as single or double "blinds", referring to the amount of information the receiving party is told in advance about them. With single blinds, the laboratory knows that the samples are for audit purposes but does not know the analytes and/or the concentrations present. With double blinds, the laboratory does not know that the samples are audit samples. Typical performance audits provide the means to assess precision and accuracy, as well as analyte identification.

A systems audit is an inspection and review of the data generation, quality control and support system of an analytical laboratory. This inspection and compliance review includes all activities related to the requirements established for the laboratory quality assurance program. Typical systems audit include an evaluation of the following:

- Assessment of degree of compliance with the Quality Assurance Program including certification programs, SOP completeness, completeness of quality assurance project plans, assessment of QA documentation, data review and approval process, internal QC program, and internal audits;
- Continuing compliance with corrective actions identified in a previous audit of the facility;
- Detailed performance audits of selected analytical programs;
- Calibration procedures and documentation;
- Sample handling procedures including chain of custody; and
- Experience of laboratory personnel.

Internal Audits

The PACE/NJ quality assurance group conducts scheduled and unscheduled audits that are designed to aid in the fulfillment of quality assurance objectives within the facility. Good laboratory practices, safety and conformance to standard operating procedures and methodologies, as well as results of internal performance audit samples, are reviewed by the QA staff. Systems audits are scheduled on a bi-monthly basis.

Reports may be submitted to the area managers and laboratory director when non-conformances are observed, or other followup is taken. The responsible area managers are accountable for the timely implementation of the corrective actions. Unscheduled audits are conducted to confirm

that critical concerns have been addressed. Corrective actions are monitored. Internal audit reports are confidential to PACE/NJ employees.

External Audits

Clients and regulatory agencies routinely audit the PACE/NJ facility. The audits include performance evaluation samples submitted as blinds or double blinds for analysis, as well as announced and unannounced on-site laboratory inspections. The QA staff hosts or otherwise participates in external systems audits, which occur frequently on an ongoing basis. The external audit schedule is variable based upon client and regulatory requirements.

The laboratory participates in a number of ongoing, scheduled performance audit activities. PACE/NJ participates twice yearly in both the USEPA Water Supply and Water Pollution performance evaluation studies, quarterly in proficiency studies from the State of New York (two potable water and two non-potable water/solid/hazardous waste studies per year) and annually in a limited study from the State of Wisconsin. United States Army Corps of Engineers (USACE) proficiency samples, required for initial and continued validation, are analyzed, at minimum, every eighteen months. In general, PACE/NJ analyzes external performance evaluation samples for organic and inorganic parameters that support approval in the environmental laboratory certification programs described below.

PACE/NJ is regularly audited by state agencies for compliance to the state's certification regulations. Private sector laboratory approval programs also include periodic laboratory audits. Projects and contracts frequently require laboratory inspection prior to award and at designated intervals thereafter. The PACE/NJ facility's managers are responsible for responding to the findings of external audits using the same mechanisms employed for internal audits, and for implementing corrective actions.

Certifications and Approvals

The PACE/NJ laboratory participates in a number of contracts and state certification programs. Approval entails laboratory evaluation which includes, but is not limited to, representative proficiency samples, initial and periodic systems audits and other proof of laboratory qualification.

It has been PACE/NJ's policy to obtain appropriate state certification for every active project. PACE/NJ currently participates in fifteen state certification programs (refer to Table 12.1). The laboratory holds state approvals for the analysis of drinking waters, non-potable waters and solid/hazardous wastes; the types of certification available to environmental laboratories varies from state to state.

PACE/NJ holds United States Army Toxic and Hazardous Materials Agency (USATHAMA), United States Army Corp of Engineers (USACE) and Naval Energy and Environmental Activity Installation Restoration Program (NEESA IRP) Department of Defense (DOD) approval for organic and inorganic parameters. The laboratory was awarded a NJDEP X-26174 Analytical Services Contract for an array of Task III (SW846) and Task IV (USEPA CLP) determinations

Data Quality

The PACE/NJ laboratory maintains records of the quality control data generated for analytical batches, including, but not limited to, method blank, spiked blank recovery, spiked sample recovery, duplicate sample, and surrogate recovery data. PACE/NJ's computer system is utilized for the statistical manipulation of these data points. The data are used to determine precision, accuracy, method validity and statistical process control, as well as to monitor performance for corrective actions.

The quality assurance group administers a program designed to investigate and resolve internal nd external data challenges. The goal is to ensure that the issues, investigations and resolutions e documented and tracked. The overall system, particularly the self-inspection aspect, enables ACE/NJ to develop strategies and policies to reduce any systematic errors.

PREVENTATIVE MAINTENANCE PROCEDURES

PACE/NJ, being a highly computerized and instrument oriented laboratory, maintains maintenance contracts with major instrument manufacturers for 24 hour, 7 days per week emergency call service. PACE/NJ performs routine maintenance to prevent instrument malfunction and minimize downtime, and to optimize instrument capabilities.

In the event of an instrument breakdown, there are several options that may be considered: schedule the work on another instrument while service is being performed; request that the vender provide an instrument to use for the interim; subcontract the work to an approved outside or PACE/NJ network laboratory (providing certification requirements are satisfied and the client consents).

Preventative Maintenance

Analysts are trained to respond to instrument maintenance needs. Criteria for this type of maintenance is based on instrument performance. Failure of instruments to perform according to stated methodologies and criteria limits drives the need for daily maintenance. The schedule of preventative or routine maintenance checks are, in general, outlined within the specific equipments' operators manuals and in the analytical procedures performed. PACE/NJ adheres to these schedules, and it is the responsibility of both the analyst and department manager to ensure these checks are completed.

Replacement Parts

The laboratory maintains an inventory of replacement parts for all analytical instrumentation. This enables PACE/NJ analysts to perform routine maintenance and repair of instruments as needed.

Record ke[illegible] and Preventative Maintenance Logbooks

Instrument spe[illegible]ogbooks are utilized to record instrument problems, maintenance and demonstration of control. PACE/NJ maintains one current logbook per instrument. Analysts are equired to record all maintenance performed on an instrument; outside service engineer records are included so the maintenance documentation is complete. If an instrument is unusable, a label stating so is placed on the instrument to avoid its use.

Thermometers Refrigerators, Ovens and Balances

Laboratory [illegible]ermometers are calibrated against NIST traceable thermometers annually. The results are recorded in a logbook specific to that purpose. Correction factors are recorded on the thermometer tags, along with the unique thermometer identification number and calibration date, and used by PACE/NJ personnel to correct actual temperature measurements. The correction factor is applied to each reading until the thermometer is calibrated again. Use of thermometers with a correction of > 5° C is avoided. PACE/NJ minimizes the need to apply corrections by utilizing the correct media, thermometers and procedures during calibration.

Refrigerators and ovens are monitored once or twice daily or as used, dependent upon the function of the unit. Logbooks are maintained by the units to record monitoring and results. If

a unit fails acceptance criteria, monitoring is co ued til the temperature stabilizes within the range or appropriate corrective actions are take.. Mon.oring occurs at one (1) hour intervals for a maximum four (4) hour period; if the reading following the temperature control adjustment is out, the unit is considered "out of order", a is emptied and serviced. It is not put back into service until shown to be stable at the requi d temperature range.

Analytical ba ces are calibrated annually by an outside service. A dated sticker, certifying the calibration, is aced on each balance. Records for balance calibrat are maintained in PACE/NJ QA files. Multi- and singl oint calibration checks . ly performed to e ure the accuracy e h balance. T sults are recorded in dedicated logbooks that are intained at each balance location. Balances that do not satisfy specifications are taken out of service for replacement or repair.

SPECIFIC ROUTINE PROCEDURES TO ASSESS DATA PRECISION AND ACCURACY

For every batch of samples analyzed, a series of quality control samples are analyzed to assess the precision, accuracy and validity of the analysis. These data are reviewed before release of the data. All QC data are stored at PACE/NJ and are useable for determination of method precision and accuracy. The laboratory utilizes its computer data base system to apply the QC routines for generation of statistics and QC charts. PACE/NJ makes every effort to meet or exceed the accuracy and precision data as defined within specific methodologies. However, for actual matrices these data may not be comparable. If no precision or accuracy requirements are specified within a methodology, PACE/NJ will establish criteria, and maintain the quality control limits for valid method use.

Accuracy is characterized by the degree of agreement of a measured value to the accepted true value. Data comparability is a fundamental underlying assumption to all accuracy assessments. Accuracy assessments are used to establish the bias present in the data. Precision is characterized by the degree of agreement of a measured value to another measured value. Data comparability is a fundamental underlying assumption to all precision assessments. Precision assessments are used to establish the control status of a system, such as a sampling process or a measurement process.

Completeness is characterized by the degree of agreement of the quality of a data set to the method and/or client specification. Completeness is defined as the percentage of meaasurements made which are judged to be valid measurements. The completeness goal is to generate a sufficient amount of valid data based on project needs. Comparability of the quality control data model to the experimental data set is a fundamental assumption to all completeness assessments. Comparability is a measure of the confidence with which one data set can be compared to another data set. Completeness assessments are used to characterize the applicability of the data.

To estimate accuracy, spiked blank samples and matrix spike sample recoveries are evaluated. This allows for the determination of both method and actual sample batch accuracy. Precision is measured and monitored in two ways: using range control for duplicate pairs and relative percent difference. PACE/NJ uses the formulas presented in Standard Methods and the USEPA Quality Assurance handbooks for calculations of precision and accuracy.

Accuracy Control

The objective of the laboratory concerning accuracy is to meet or exceed the accuracy criteria specified in an analytical method. Accuracy determinations are performed for each parameter according to the specifications of the particular method employed. Accuracy assessments are performed by the analysts via a first level data review. The analysts will compare data results to the established acceptance criteria. When the criteria is not met additional characterization of the data is required according to the requirements of the methodology and as determined by the judgement of the analyst in order to establish the accuracy of the data.

For each type of spiked sample accuracy control charts are developed. Control limits are established according to the requirements of the methodology. In the absence of published control criteria the limits are calculated. The limits are calculated based on the mean and

standard deviation of a pooled data set. The data set must contain of no less than seven (7) data results. Limits are then calculated.

Precision Control

The objective of the laboratory concerning precision is to meet or exceed the criteria specified in an analytical methodology. Precision measurements are performed for each parameter according to the specifications of the particular method employed. Precision assessments are performed by the analysts via a first level data review. The analysts will compare data results to the established acceptance criteria. When the criteria is not met then additional characterization of the data is required according to the requirements of the methodology and as determined by the judgement of the analyst in order to establish the precision of the data.

For each type of replicate sample and MS/MSD pair, precision control charts are developed. An upper control limit is established according to the requirements of methodology. In the absence of published control criteria, the limit is established as 20 percent.

Calculations To Determine Accuracy

Accuracy is calculated as follows.

$$\% \text{ Recovery} = \frac{(X - T)}{K} \times 100$$

where:
- K Known addition of the spiked compound
- X Analytical result from the spiked sample
- T Analytical result from the unspiked aliquot

Standard deviation (Sp) is used for determining the variation among several recovery samples, and establish upper and lower warning and control limits. Standard deviation is calculated as follows.

$$Sp = \text{the square root of } \frac{(X - \bar{X})^2}{(n - 1)}$$

where:
- Sp Standard deviation of % Recovery
- X Observed value
- $\bar{X}$ Mean or average of all observations
- n Number of observations

Control limits are created to determine the acceptable range of analyte recovery and are used to compare actual spike recovery results against. Control limits are calculated as follows.

$$UCL = \bar{X} + 3\ Sp$$

$$LCL = \bar{X} - 3\ Sp$$

$$UWL = \bar{X} + 2\ Sp$$

$$LWL = \bar{X} - 2\,Sp$$

where:
- $\bar{X}$ Average Value
- Sp Standard Deviation
- UCL Upper Control Limit
- LCL Lower Control Limit
- UWL Upper Warning Limit
- LWL Lower Warning Limit

Calculations To Determine Precision

The precision of duplicate sample pairs is expressed as Relative Percent Difference (RPD) and is calculated as follows.

Relative Percent Difference

$$RPD = \frac{|A - B|}{(A + B)/2} \times 100$$

where:
- RPD Relative Percent Difference
- A Replicate value 1
- B Replicate value 2

Standard deviation can be used to determine variation among several RPD values for duplicate pairs and establish statistical limits for duplicate RPD. Standard deviation and control limit calculations are shown in the above discussion of accuracy. Range control may also be used.

Range Control

$$R = A - B$$

$$\bar{X} = \frac{A + B}{n}$$

where:
- R Range of a pair of results
- X Average of a pair of results
- A Duplicate value 1
- B Duplicate value 2
- n n = 2 represents a single duplicate pair

To graphically represent the data of numerous duplicate pairs on control charts, the following calculations are performed using statistical numbers.

$$\bar{\bar{X}} = \text{the sum of } \bar{X} / n$$

$$\bar{R} = \text{the sum of } R / n$$

where:

$\bar{\bar{X}}$	Grand Mean
$\bar{R}$	Average Range
X	Average of a pair of results
R	Range of a pair of results
n	n = 2 represents a single duplicate pair

Control limits for ranges (R - bar chart):

$$CL = 3.27\ (\bar{R})$$

$$WL = \bar{R} \pm 2/3\ (3.27\ \bar{R} - \bar{R})$$

where:

$\bar{R}$	Average Range
CL	Control Limit
WL	Warning Limit

To determine if the proper range control chart is being used for evaluation of a duplicate pair of results, the X control chart may be used.

Control limits for averages (X - bar chart):

$$UCL = \bar{\bar{X}} + 1.88\ (\bar{R})$$

$$LCL = \bar{\bar{X}} - 1.88\ (\bar{R})$$

$$UWL = \bar{\bar{X}} + 2/3\ (1.88\ \bar{R})$$

$$LWL = \bar{\bar{X}} - 2/3\ (1.88\ \bar{R})$$

where:

$\bar{\bar{X}}$	Grand Mean
R	Mean Range
UCL	Upper Control Limit
LCL	Lower Control Limit
UWL	Upper Warning Limit
LWL	Lower Warning Limit

Standard deviation provides the basis for the determination of precision from pooled spiked sample accuracy data. This precision determination may be used to establish control limits as described in the accuracy discussion above.

Calculation To Determine Completeness

The percent completeness obtained for a data set is calculated as folows.

$$\% \text{ Completeness} = \frac{\text{Number of control parameters that satisfy criteria}}{\text{Total number of control parameters}} \times 100$$

CORRECTIVE ACTION

Corrective action is defined as those actions necessary to place any operational process or analytic back into its performance specification. It is the objective of the laboratory to implement appropriate corrective action when and where necessary in an effective and timely manner.

When a situation or issue is identified that requires a corrective action, it is investigated and evaluated using processes that address the requirements for the specific non-conformance found. In a case where there is a lack of existing requirements that may be applied, identification of the requirements is included in the corrective action plan.

The corrective action is identified and documented through one of several internal report processes. The Investigation/Correction Log (Figure 13.1) is utilized for internal and external issues, client and agency inquiries, performance and systems audits, and other non-routine occurrences, findings or recognized needs. A Routine Corrective Action Log (Figure 13.2) is produced for every batch of analytical samples and used by the technical staff prior to reporting sample data. The form is used to document routine analytical problems and return-to-control following corrective action activities described in the methods and PACE/NJ SOPs.

In the event that any corrective actions are needed because the data quality is in question above and beyond those actions stipulated in the analytical methods, the client will be contacted by PACE/NJ to discuss the problem.

Corrective actions are initiated at all operational levels within the laboratory, involving analysts, their management, and the quality assurance group in both formal and informal procedures. Analysts are responsible for taking routine informal corrective actions described in the methods. Corrective actions are also initiated externally through project management personnel in local, state, or federal agencies and private sector clients. In each case, after an assessment of the issue, appropriate steps are taken to correct the situation and prevent it from occurring again.

Depending on the severity, corrective actions may be taken at the analyst level, department level, or within the entire laboratory. PACE/NJ recognizes the importance of corrective action to maintain a high quality program. In this light, data are reviewed for completeness, accuracy, precision and compliance with analytical method quality control and project specifications during the data generation and reporting process.

Specific corrective action responses are performed for quality assurance/quality control deficiencies; many are pre-defined in the analytical protocol used for the samples. In general, there are three major types of corrective actions which are initiated at PACE/NJ.

To correct sample problems: Individual samples or matrix problems are usually handled within the analytical laboratory. Corrective actions may include repreparation and re-analysis, clean-ups, dilutions or matrix modifications. All actions taken are documented with the analytical results.

A typical example is when organic surrogate compound recoveries are outside of the acceptance limits; if no calculation or other problems are found, the sample is routinely reprepared/reanalyzed to verify matrix effects. Every effort is made to meet sample hold times.

To correct sample batch problems: An entire batch of samples may require corrective action if quality control criteria are not met. Laboratory managers are involved in the decisions for actions which may include re-analysis, re-extraction, etc. The quali ssurance staff may review both sets of data as applicable to determine the problems have bee resolved.

A typical example is when method blank contamination occurs; if analytes are conti present a unacceptable levels, the batch is r reprepared/re zed after the ource of amination has been tified and eliminated. Every effort is made to meet sample hold times.

To correct systematic problems: Those problems of a p edural nature ar d by the laboratory managers and quality assurance group. Major operational changes are made only after approval of the Quality Assurance and Laboratory Operations Managers.

Systematic problems are identified as repetitive in nature or involving number of samples or batches. Procedures involving analyst technique or training, or use of defective equipment or materials are identified and corrected.

QUALITY ASSURANCE REPORTS TO MANAGEMENT

The objective of the PACE/NJ quality assurance program is to ensure that an operational system is in place which enables management to determine the quality level of all data produced within the laboratory system. An essential component of the system is the communication pathways and feedback mechanisms which insure that management obtains quality information promptly and consistently. To achieve this objective, PACE/NJ employs informal and formal reporting processes to ensure that management is informed of operational quality. This information enables PACE/NJ to take corrective action promptly when required. Reporting occurs at the following frequency.

- Daily meetings to discuss possible quality assurance problems and proposed solutions.
- Weekly meeting with upper management to discuss laboratory performance, upcoming audits, certification programs, and past audit performances.
- Monthly written status reports to upper management; inclusion of all quality assurance programs and pertinent laboratory issues.
- As required, internal departmental audit reports with observations and suggested corrective action procedures.

APPENDIX -

FIGURES AND TABLES

Figure 1.1

PACE delivers...

The Right Chemistry, The Right Solution.

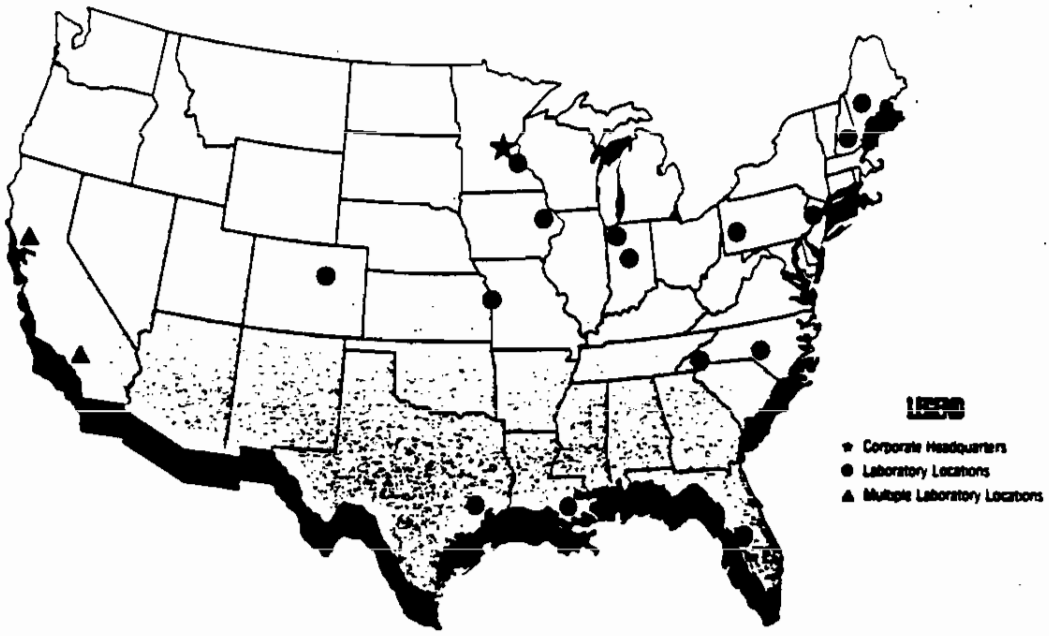

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Figure 3.1

Ethical Conduct Agreement

I. I understand the high ethical standards required of me with regard to the duties I perform and the data I report in connection with my employment at ETC.

II. I have received formal instruction on the code of ethics that has been adapted by ETC and agree to comply with these requirements.

III. I also agree to the following:

a. I shall not intentionally report data values which are not the actual values observed or measured.

b. I shall not intentionally modify data values unless the modification can be technically justified through a measurable analytical process.

c. I shall not intentionally report dates and times of data analysis that are not the true and actual times the data analysis was conducted.

d. I shall not condone any accidental or intentional reporting of inauthentic data by other employees and immediately report it's occurrence to my superiors.

e. I shall immediately report any accidental reporting of inauthentic data by myself to my superiors.

_____

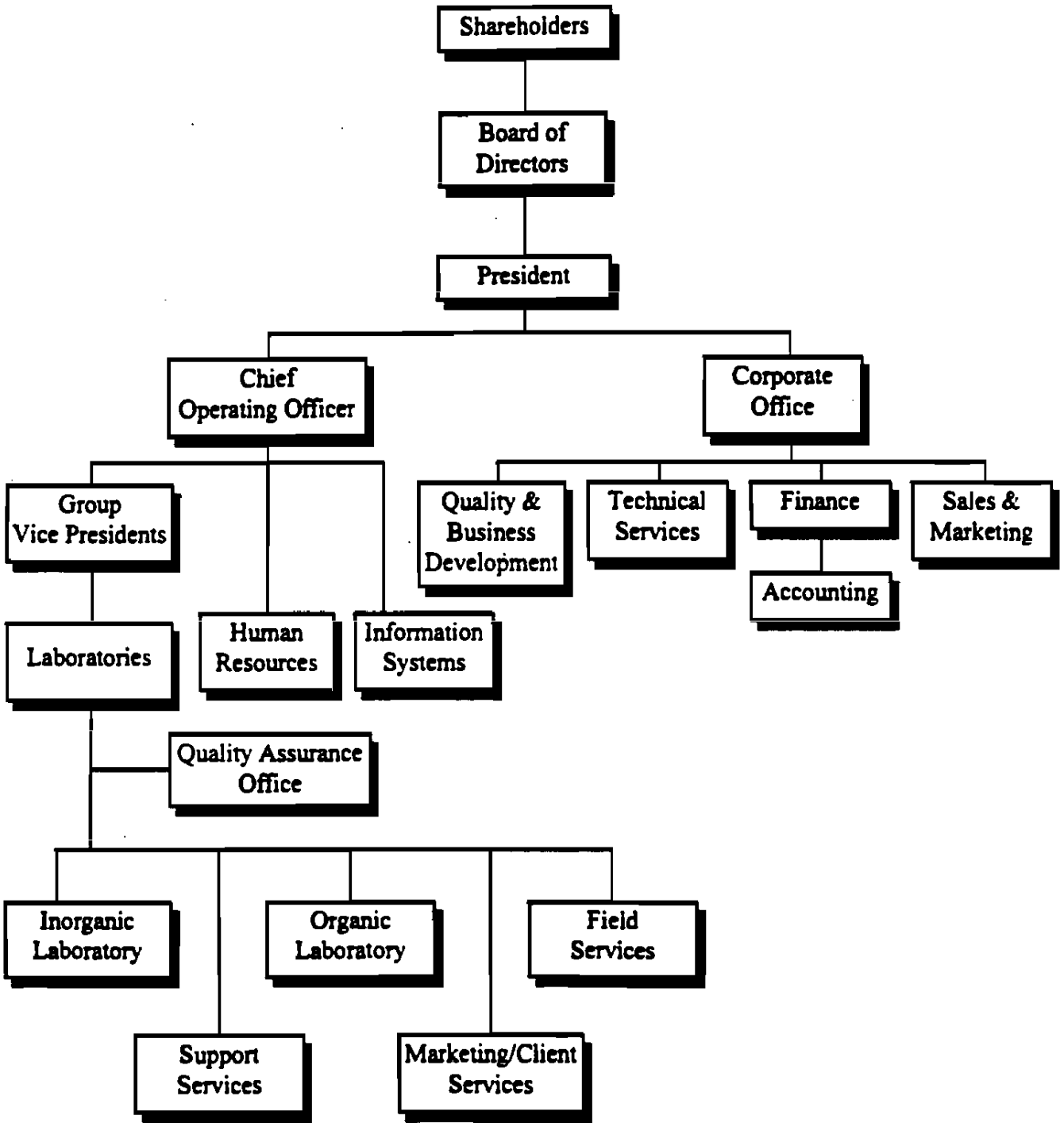
(Signature)

_____

(Date)

Figure 4.1

PACE Organizational Structure



PACE/NEW JERSEY
August 1994

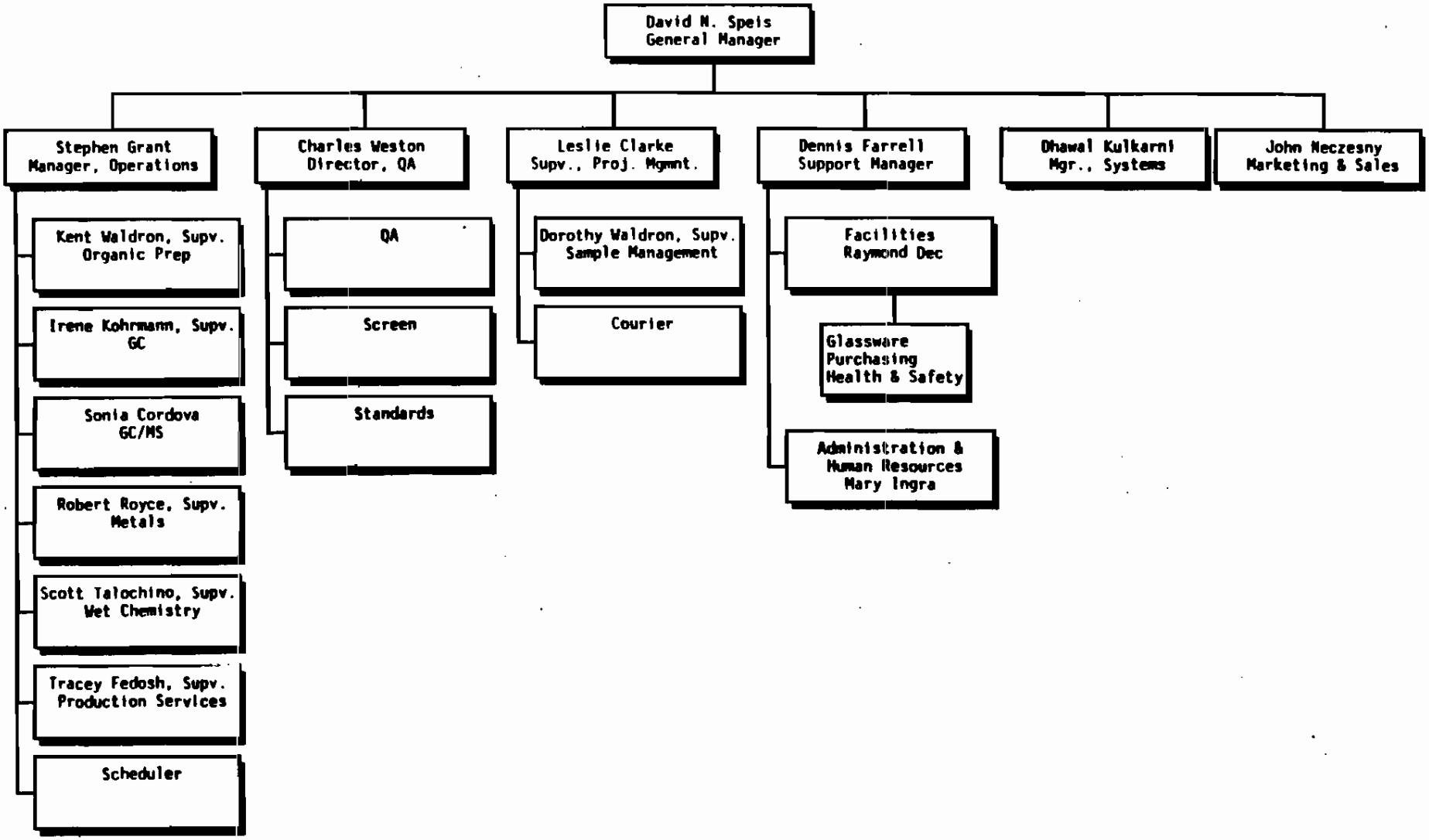


Figure 4.2

Figure 4.3

Project Organization - PACE/NJ Personnel

Title	Name*
Account Executive	
Technical Project Manager	
Operations Manager	
Quality Assurance Officer	

* Will be designated on a by-project basis.

Table 5.1 PACE/NJ Matrix Spike Percent Recovery Limits

Fraction	Parameter	Water	Soil
Volatile Organics by GC/MS			
VOA	1,1-Dichloroethene	61-145	59-172
VOA	Trichloroethene	71-120	62-137
VOA	Chlorobenzene	75-130	60-133
VOA	Toluene	76-125	59-139
VOA	Benzene	76-127	66-142
Extractable Organics by GC/MS			
BN	1,2,4-Trichlorobenzene	39-98	38-107
BN	Acenaphthene	46-118	31-137
BN	2,4-Dinitrotoluene	24-96	28-89
BN	Pyrene	26-127	35-142
BN	N-Nitroso-Di-n-Propylamine	41-116	41-126
BN	1,4-Dichlorobenzene	36-97	28-104
Acid	Pentachlorophenol	09-103	17-109
Acid	Phenol	12-110	26-90
Acid	2-Chlorophenol	27-123	25-102
Acid	4-Chloro-3-Methylphenol	23-97	26-103
Acid	4-Nitrophenol	10-80	11-114
PCDX	Polychlorinated Dibenzo-p-Dioxins/Furans	60-150	60-150
Extractable Organics by GC			
Pest	Lindane	56-123	46-127
Pest	Heptachlor	40-131	35-130
Pest	Aldrin	40-120	34-132
Pest	Dieldrin	52-126	31-134
Pest	Endrin	56-121	42-139
Pest	4,4'-DDT	38-127	23-134
Inorganics			
	Metals	75-125	75-125
	Wet Chemistries	75-125	75-125

These are USEPA CLP advisory limits, with the exception of PCDX and the wet chemistries.

Matrix spike recoveries may be highly affected by the nature of the sample spiked. Mild to severe matrix interferences are frequently encountered with environmental samples; therefore, results outside of the limits may simply demonstrate the effect of the matrix on analyte recovery given the limitations of the methods performed.

Limits are not soley used to determine if a sample should be reanalyzed; matrix spike recovery contributes to the overall quality control assessment of analytical data and any subsequent corrective actions taken by the laboratory as a result of its evaluation.

Table 5.2 PACE/NJ Duplica' Relative Percent Diff' ence (RPD) Limits

Fraction	Parameter	Water	Soil
Volatile Organics GC/MS			
VOA	1. ichloroethene	14	
VOA	T. loroethene	14	24
VOA	Chlorobenzene	13	21
VOA	Toluene	13	21
VOA	Benzene	11	21
Extractable Organics by G MS			
BN	1,2,4-Trichlorobenzene	28	23
BN	Acenaphthene	31	19
BN	2,4-Dinitrotoluene	38	47
BN	Pyrene	31	
BN	N-Nitroso-Di-n-Propylamin	38	
BN	1,4-Dichlorobenzene	28	27
Acid	Pentachlorophenol	50	47
Acid	Phenol	42	35
Acid	2-Chlorophenol	40	50
Acid	4-Chloro-3-Methylphenol	42	33
Acid	4-Nitrophenol	50	50
PCDX	Polychlorinated Dibenzo-p-Dioxins/Furans	50	50
Extrac..ole Organics by GC			
Pest	Lindane	15	50
Pest	Heptachlor	20	31
Pest	Aldrin	22	43
Pest	Dieldrin	18	38
Pest	Endrin	21	45
Pest	4,4'-DDT	27	50
Inorganics			
	Metals (a)	20	20
	Cyanides (a)	20	20
	Wet Chemistries (b)	25	25

These are USEPA CLP RPD advisory limits, with the exception of PCDX and the wet chemistries.

The RPD of duplicat vironmental samples may be highly affected by the natur samp matrix. Mild t. ere heterogeneity is frequently encountered within a sam s may result in variable matrix interferences or concentrations of analytes inherently p nt. Therefore, elevated RPDs may simply demonstrate the effect of the matrix on precision given the limitations of the me.ods performed.

Limits are not soley used to determine if a sample should be reanalyzed; the RPD contributes to the overall quality control assessment of analytical data and any subsequent corrective a s taken by the laboratory as a result of its evaluation.

Footnotes:
(a) +/- CRDL WHEN <5X CRDL; +/-20% WHEN >5X CRDL
(b) +/- 50% WHEN <5X MDL; +/- 25% WHEN >5X MDL

Table 5.3 PACE/NJ Surrogate/System Monitoring Compound (SMC) Recovery Limits Organic Analyses

Fraction	Surrogate/SMC Compound	Water	Low/Med Soil
SW846, 600s, 2/88 CLP			
Volatile (GC/MS)	Toluene-d_8	88-110	81-117
	4-Bromofluorobenzene	86-115	74-121
	1,2-Dichloroethane-d_4	76-114	70-121
Volatile (GC-VHC)	1,4-Dichlorobutane	80-120	80-120
Volatile (GC-VAr)	a,a,a-Trifluorotoluene	80-120	80-120
Acid (GC/MS)	Phenol-d_5	10-94	24-113
	2-Fluorophenol	21-100	25-121
	2,4,6-Tribromophenol	10-123	19-122
Base/Neutral (GC/MS)	Nitrobenzene-d_5	35-114	23-120
	2-Fluorobiphenyl	43-116	30-115
	Terphenyl-d_{14}	33-141	18-137
Pest/PCB (GC-ECD)	Dibutylchlorendate	24-154 (a)	20-150 (a)
	Tetrachloro-m-xylene	46-185	34-160
Pesticide (GC-FPD)	Ethyl-p-nitrophenyl-benzenethiophosphonate	24-154	20-150
Herbicide (GC-ECD)	Dicamba	03-143	03-143
PCDX (GC/MS)	13C-isomers (b)	10-120	10-120
3/90 CLP, OLM01			
Volatile (GC/MS)	Toluene-d_8	88-110	84-138
	4-Bromofluorobenzene	86-115	59-113
	1,2-Dichloroethane-d_4	76-114	70-121
Acid (GC/MS)	Phenol-d_5	10-110	24-113
	2-Fluorophenol	21-110	25-121
	2,4,6-Tribromophenol	10-123	19-122
	2-Chlorophenol-d_4	33-110 (a)	20-130 (a)
Base/Neutral (GC/MS)	Nitrobenzene-d_5	35-114	23-120
	2-Fluorobiphenyl	43-116	30-115
	Terphenyl-d_{14}	33-141	18-137
	1,2-Dichlorobenzene-d_4	16-110 (a)	20-130 (a)
Pesticides (GC)	Decachlorobiphenyl	60-150 (a)	60-150 (a)
	Tetrachloro-m-xylene	60-150 (a)	60-150 (a)

Footnotes:

(a) Advisory limits; not soley used to determine if a sample should be reanalyzd.

(b) Internal Standards (IS) are added at preparation for Polychlorinated Dibenzo-p-Dioxins and Polychlorinated Dibenzofurans analyses. The recovery standard is added at analysis to quantitate the IS concentration, and recoveries for the IS are calculated. The IS compounds are listed on this table as functional surrogate compounds for the method performed.

Table 6.1 Containers, Holding Times and Preservatives

Parameter	Container (a)	Holding Time (b)	Preservative (c)
Wet Chemistry, Standard Analyses			
Total Organic Carbon (TOC)	1 x 125 ml, g	28 days	H_2SO_4, pH < 2; Cool, 4° C
Total Organic Halides (TOX; dupl/quadruplicate)	1 x 500 ml; or 2 x 500 ml, g (d)	28 days	H_2SO_4, pH < 2; $NaSO_3$
Chemical Oxygen Demand (COD)	1 x 125 ml, g	28 days	H_2SO_4, pH < 2; Cool, 4° C
Total Petroleum Hydrocarbons	1 x 1 L, g	28 days	H_2SO_4, pH < 2; Cool, 4° C
Cyanide, Total (in-line/manual distillation)	1 x 125 ml; or 1 x 1 L, g	14 days	NaOH, pH > 12; Cool, 4° C
Phenolics, Total (in-line/manual distillation)	1 x 125 ml; or 1 x 500 ml, g	28 days	H_2SO_4, pH < 2; Cool, 4° C
Sulfate	1 x 125 ml, g	28 days	Cool, 4° C
Chloride	1 x 125 ml, g	28 days	Cool, 4° C
Nitrate as N	1 x 125 ml, g	28 days	H_2SO_4, pH < 2; Cool, 4° C
Nitrite as N	1 x 125 ml, g	28 days	H_2SO_4, pH < 2; Cool, 4° C
Fluoride	1 x 125 ml, g	28 days	Cool, 4° C
Specific Conductance	1 x 500 ml, g	28 days	Cool, 4° C
Total Solids	1 x 500 ml, g	7 days	Cool, 4° C
Total Dissolved Solids	1 x 500 ml, g	7 days	Cool, 4° C
Total Suspended Solids	1 x 500 ml, g	7 days	Cool, 4° C
Sulfide	2 x 125 ml, g (d)	7 days	Zn acetate & NaOH, pH > 9; Cool, 4° C
Inorganics, Standard Analyses			
Metals, except Mercury	1 x 1 L, p or g	6 months	HNO_3, pH < 2; Cool, 4° C
Mercury	1 x 1 L, g	28 days (e)	HNO_3, pH < 2; Cool, 4° C
Organics, Standard Analyses			
Volatiles, purge & trap (GC/MS)	3 x 40 ml, g (d)	14 days (f)	HCl, pH < 2; Cool, 4° C
Volatiles, heated purge & trap (GC/MS)	3 x 40 ml, g (d)	14 days (f)	HCl, pH < 2; Cool, 4° C

Table 6.1 Continued

Parameter	Container (a)	Holding Time (b)	Preservative (c)
Purgeable Aromatics (GC)	2 x 40 ml, g (d)	14 days (f)	HCl, pH < 2; Cool, 4° C
Purgeable Halocarbons (GC)	2 x 40 ml, g (d)	14 days	Cool, 4° C
Semivolatiles, Acid/base/neutral (GC/MS)	2 x 1 L, g	7/14 days (prep); 40 days (analysis)(g)	Cool, 4° C
Pesticides and/or Aroclors (GC, GC/MS)	2 x 1 L, g	7/14 days (prep); 40 days (analysis)(g)	Cool, 4° C
Herbicides (GC)	2 x 1 L, g	7/14 days (prep); 40 days (analysis)(g)	Cool, 4° C
Polychlorinated Dibenzo-p-Dioxins/Furans (GC/MS)	2 x 1 L, g (water)	7 days (prep); 40 days (analysis)(g)	Cool, 4° C
	1 x 500 ml, g (soil)	30 days (prep); 45 days (analysis)	
Polynuclear Aromatic Hydrocarbons (HPLC)	2 x 1 L, g	7/14 days (prep); 40 days (analysis)(g)	Cool, 4° C
Toxicity Characteristic Leaching Procedure (k) Preparation of the TCLP Extract			
Volatiles	1 x 125 ml, g (d)	14 days	Cool, 4° C (h)
Semi-volatiles	1 x 1L, g	14 days	Cool, 4° C (h)
Metals, except Mercury	1 x 1L, p or g	180 days	Cool, 4° C (h)
Mercury	1 x 1L, p or g	28 days	Cool, 4° C (h)
Toxicity Characteristic Constituents Analysis of the TCLP Extract			
Volatiles, purge & trap	3 x 40 ml, g (d)	14 days (i)	HCl, pH < 2; Cool, 4° C
Semi-volatiles, Acid/base/neutral, pesticides	2 x 1 L, g (d)	7 days (prep)(i) 40 days (analysis)(g)	Cool, 4° C
Semi-volatiles, Herbicides	2 x 1 L, g (d)	7 days (prep)(i) 40 days (analysis)(g)	Cool, 4° C
Metals, except Mercury	1 x 1 L, p	180 days (i)(j)	HNO_3, pH < 2 (j)
Mercury	1 x 1 L, g	28 days (i)(j)	HNO_3, pH < 2 (j)
U.S. EPA CLP Parameters (l)			
Volatile Organics	3 x 40 ml; g (d)	10 days	HCl, pH < 2; Cool, 4° C
Semivolatile Organics (BNA)	2 x 1 L; g	5/10 days (prep); 40 days (analysis)(g)	Cool, 4° C

Table 6.1 Continued

Parameter	Container (a)	Holding Time (b)	Preservative (c)
Semivolatile Organics (Pest/PCB)	2 x 1 L; g	5/10 days (prep); 40 days (analysis)(g)	Cool, 4° C
Metals (except Mercury)	1 x 1 L; p	6 months	HNO_3, pH < 2; Cool, 4° C
Mercury	1 x 1 L; p	26 days	HNO_3, pH < 2 Cool, 4° C
Cyanide, Total	1 x 1 L, g	12 days	NaOH, pH > 12; Cool, 4° C

Notes: g = glass; p = plastic

Sulfide is a subcontract parameter.

Source: 40 CFR Part 136 Table II; SW-846 3rd Edition, Revision 0 and I; U.S. EPA CLP Statements of Work.

Footnotes:

(a) Minimum volume requirements for aqueous samples are listed for extractable organic parameters by GC and GC/MS methods. An additional two (2) liters are required for matrix spike and matrix spike duplicate preparation for each analytical method when the sample is utilized for QC purposes.

Lesser sample amounts may be required for soil/sediment samples.

(b) Hold time from date of sampling unless otherwise noted; Aqueous/Non-aqueous where applicable.

(c) Chemical preservation is not applicable to non-aqueous matrices. Premeasured amounts of preservatives are supplied in small ampules attached to the proper bottles for water matrices.

(d) Filled with minimal headspace to prevent analyte loss.

(e) For SW846, 13 days if container is plastic.

(f) If pH > 2, hold time is shortened to 7 days.

(g) Hold time from date of preparative extraction.

(h) No chemical preservatives should be added to samples prior to TCLP extraction; samples may be cooled as indicated unless refrigeration results in irreversible physical changes to the sample. The laboratory should be informed if precipitation occurs so that the entire sample, including the precipitate, may be extracted.

(i) Hold time from completion date of TCLP extract preparation.

(j) If precipitation is observed upon addition of nitric acid to a small aliquot of the TCLP extract, the remaining portion will not be acidified and the extract will be analyzed as soon as possible.

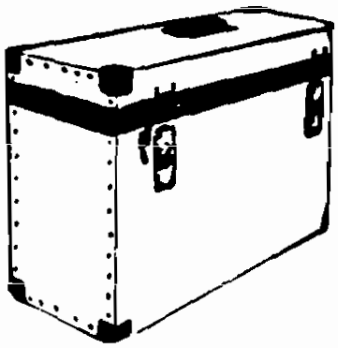
(k) Minimum estimated volume requirements for the intact sample are listed for the leaching procedure. Additional sample may be required depending on the physical nature of the sample and/or matrix spike preparation. Should this be the case, the laboratory will contact the client immediately for resolution. The laboratory will send extra bottles for the initial sampling event if there is reason to believe or concern that the volumes stated will not satisfy the mass (weight) requirements of the method.

(l) Hold time from verified time of sample receipt (VTSR) at the laboratory.

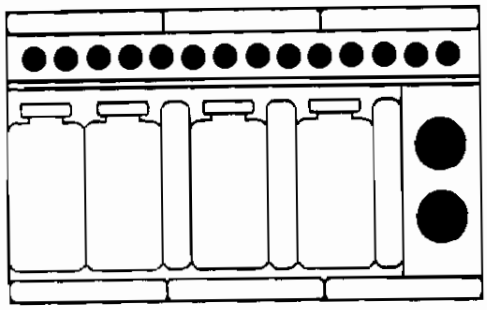
Figure 6.1 PACE/NJ Sample Shuttle™

The PACE/NJ Shuttle™ is a uniquely engineered and patented shipping container that has been designed to meet and exceed USEPA and State protocols for shipping, chemical and thermal preservation of analytical samples. The container can be locked and sealed to ensure sample integrity during both transport and sampling.

Diagram of Shuttle & Example Bottle Configuration



Exterior



Interior - Cross Section
Example Configuration

Features Include:

A convenient suitcase shape for ease of transportation to and from sampling locations;

A rugged waterproof outer container that withstands varying field conditions;

Polyurethane foam insulation for dependable thermal performance;

Chain of Custody locks and numbered seals preserve sample integrity;

Customized coolant to sustain required EPA temperature controls;

Chain of Custody and Field Parameter forms to document sampling event;

Polyethylene modular sleeve designed to prevent bottle breakage during shipment;

Pre-labeled, pre-configured bottles specific for each sampling event;

Pre-measured preservatives are attached to bottles with color coded instructions to ensure proper protocols are followed; and

Return shipping labels and custody seals to provide rapid correct return of samples to the lab.

Figure 6.1 continued

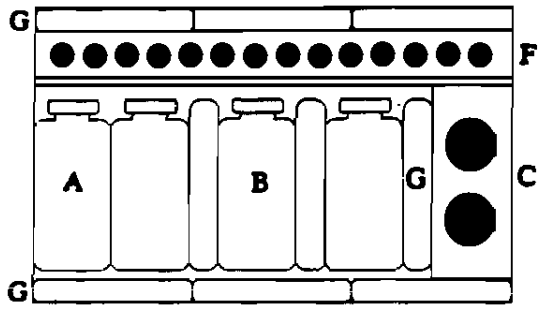
Sample Shuttle Instructions
Please Read Carefully Before Sampling

1. Freeze the ice packs at least 24 hours before sampling if the ice packs are requested from the laboratory in their unfrozen state.

2. Break the black seal on the shuttle hasp. Open the shuttle and remove the plastic bag which contains: Chain-of-Custody form, Field Information form, return shipping label, clear blank shuttle seals (for temporary custody), one black numbered shuttle seal (for resealing the shuttle after sampling).

3. Examine the Chain-of-Custody and Field Information forms carefully. These must be filled out and returned for accurate processing of the samples. Note any relevant information on the forms. Please call the PACE/NJ Technical Project Manager with any questions.

4. Unpack the bottles and frozen/unfrozen ice packs. Note the order of packing. The shuttle should be repacked exactly as received. This prevents bottle breakage and/or sample freezing from occurring.



A) 1 L Plastic Bottle
B) 1 L Amber Glass Bottle
C) 125 ml Amber Glass Bottle
D) 500 ml Plastic Bottle
E) 500 ml Amber Glass Bottle
F) 40 ml Glass Vial
G) Ice Packs

Example C[illegible]ration

5. Each bottle is labeled with an PACE/NJ sample number (ie., FF3463) and the analysis to be performed (ie., TOC, VOA). Match the type of analysis and quantity of bottles against the Chain-of-Custody form. Be sure that all bottles used for a single sample point have the same PACE/NJ number. Chemical preservative solutions are in ampules attached to the bottles that will require chemical preservation.

6. After sampling and repacking the shuttle, place all completed forms into the plastic bag and place the bag into the shuttle; close the lid.

7. Apply the return shipping label on the outside of the shuttle.

8. Insert the black custody seal through one of the outside shuttle latches and lock it.

9. When Emergency or Express analysis has been requested or is found to be needed, please call the PACE/NJ Technical Project Manager and give the following shipping information: a) Carrier, b) Airbill number, c) Arrival Date, and d) Number of shuttles.

10. When anticipated sampling or send dates change, please call the PACE/NJ Technical Project Manager so that the laboratory can most accurately schedule for the samples.

Sampling Notes

For TOX and VOA samples, fill ti[illegible]e leaving no headspace (no air bubbles).

Add the ampulated acid preservative solutions to the bottles after sampling.

Indicate on the Chain-of-Custody form whether the sample has been filtered.

ENVIRONMENTAL TESTING and CERTIFICATION

Seal No. _____ ETC Job # _____

CHAIN OF CUSTODY FORM (CC1)

ORIGINAL

Date Sealed _____ By: _____

Figure 7.1

Company: _____ Attn.: _____

City/Site: _____ Phone: _____

Address: _____

SAMPLE IDENTIFICATION

Facility: _____ (Facility/Site Code) _____ (Optional Sample Point Descriptions)

Sample Point: _____ - _____ _____ _____ _____ _____

Source Code (from below) | Your Sample Point ID (left justify) | Start Date (YY/MM/DD) | Start Time (2400 hr. clock) | Elapsed Hours (composite)

Source Codes:

Well ..(W) Outfall.....(O) Bottom Sediment ....(B) Surface Impoundment....(I) Leachate Collection Sys.....(C) Other ..... (X)

Soil (S) River/Stream..(R) Generation Point ....(G) Treatment Facility .....(T) Lake/Ocean .....(L) Specify _____

SHUTTLE CONTENTS

BOTTLE				ANALYSIS	SAMPLER		LAB
No	Type	Size	Preserv.		Filt. (Y/N)	Observations	Observations

Pace Incorporated
New Jersey Laboratory
284 Raritan Center Parkway
P. O. Box 7808
Edison, NJ 08818-7808

CHAIN OF CUSTODY CHRONICLE

1. Shuttle Opened By: (print) _____ Date: _____ Time: _____
   Signature: _____ Seal #: _____ Intact: _____

2. I have received these materials in good condition from the above person.
   Name: _____ Signature: _____
   Date: _____ Time: _____ Remarks: _____

3. I have received these materials in good condition from the above person.
   Name: _____ Signature: _____
   Date: _____ Time: _____ Remarks: _____

4. Shuttle Sealed By: (print) _____ Date: _____ Time: _____
   Signature: _____ Seal #: _____ Intact: _____

LAB USE ONLY Opened By: _____ Date: _____ Time: _____

SHUTTLE # _____ TEMP. °C _____ SEAL # _____ COND. _____

No. 99 15 Rev. 11/91

Environmental Testing and Certification Corp. • 284 Raritan Center Parkway • Edison, NJ 08818 • 908-225-6700

COMPANY	FACILITY/SITE
PROJECT CONTACT	TELEPHONE NO

ITEM NO	ETC JOB #	SOURCE CODE	SAMPLE POINT	START DATE YY/MM/DD	START TIME
1					
2					
3					
4					
5					
6					
7					
8					
9					

Source Codes:
Well (W) Outfall (O)
Soil (S) River/Stream (R)
Bottom Sediment (B)
Generation Point (G)
Surface Impoundment (I)
Treatment Facility (T)
Leachate Collection Sys (C)
Lake/Ocean (L)
Other (X)
Specify

NUMBER OF CONTAINERS

FILT. (Y/N)
PRESERV.
ANALYSIS REQUIRED

LEGEND FOR PRESERVATIVE
0 - NONE
1 - HCL
2 - HNO_3
3 - H_2SO_4
4 - $Na_2SO_3 + H_2SO_4$

SAMPLER OBSERVATIONS	LAB OBSERVATIONS

CHAIN OF CUSTODY CHRONICLE

1. Shuttle Opened By (print) _____ Date: _____ Time: _____
   Signature: _____ Seal #: _____ Intact: _____
2. I have received these materials in good condition from the above pers
   Name: _____ Signature: _____
   Date: _____ Time: _____ Remarks: _____
3. I have received these materials in good condition from the above person
   Name: _____ Signature: _____
   Date: _____ Time: _____ Remarks: _____
4. Shuttle Sealed By (print) _____ Date: _____ Time: _____
   Signature: _____ Seal #: _____ Intact: _____

LAB USE ONLY Opened By: _____ Date: _____ Time: _____
SHUTTLE # _____ TEMP. ° C _____ SEAL # _____ COND. _____

REMARKS

SAMPLER'S SIGNATURE

Free Incorporated
New Jersey
284 Raritan Center Parkway
P.O. Box
Edison, NJ 08818-7808

Figure 7.2

LAB COPY

ENVIRONMENTAL TESTING and CERTIFICATION

FIELD PARAMETER FORM (CC2)

Figure 7.3

ETC JOB # _____

Sample Point [ ] [ ] [ ] [ ] [ ] [ ] [ ] [ ] [ ] [ ]
Source Code | Sample Point I.D.

FIELD PROCEDURES

PURGE DATE (YY MM DD)	START PURGE (2400 Hr Clock)	ELAPSED HRS	WATER VOL. IN CASING (Gallons)	VOLUME PURGED (Gallons)
[ ] [ ] [ ] [ ] [ ] [ ]	[ ] [ ] [ ] [ ]	[ ] [ ] [ ]	[ ] [ ] [ ] [ ]	[ ] [ ] [ ] [ ]

SAMPLING METHOD: _____

Sampler Type [ ] A-Submersible Pump, B-ISCO, C-Bladder Pump, D-Dipper/Bottle, E-Bailer, F-Scoop/Shovel, X-Other _____ (SPECIFY OTHER)

Sampler Material [ ] A-Teflon, B-Metal, C-PVC, D-Plastic, X-Other _____ (SPECIFY OTHER)

Tubing Material [ ] A-Teflon, B-Tygon, C-Polyethylene, D-Silicon, X-Other _____ (SPECIFY OTHER)

Sample Composited [Y/N] _____
Procedure/Proportions

FIELD MEASUREMENTS

Well Elevation (ft/msl) [ ] [ ] [ ] [ ] [ ] [ ]
Depth to Ground water (ft) [ ] [ ] [ ] [ ] [ ] [ ]
Groundwater Elevation (ft msl) [ ] [ ] [ ] [ ] [ ] [ ]

Well Depth (ft) [ ] [ ] [ ] [ ]
Sample Depth (non-well) (ft) [ ] [ ] [ ] [ ]

	ph	(STD)	spec. cond.	um/cm at 25° C	(other parameter)	value	units
1st	[ ] [ ] [ ] [ ]	(STD)	[ ] [ ] [ ] [ ] [ ]	um/cm at 25° C	_____	[ ] [ ] [ ] [ ]	[ ]
2nd	[ ] [ ] [ ] [ ]	(STD)	[ ] [ ] [ ] [ ] [ ]	um/cm at 25°C	_____	[ ] [ ] [ ] [ ]	[ ]
3rd	[ ] [ ] [ ] [ ]	(STD)	[ ] [ ] [ ] [ ] [ ]	um/cm at 25°C	_____	[ ] [ ] [ ] [ ]	[ ]
4th	[ ] [ ] [ ] [ ]	(STD)	[ ] [ ] [ ] [ ] [ ]	um/cm at 25°C	_____	[ ] [ ] [ ] [ ]	[ ]

[ ] [ ] [ ] [ ] (°C) Sample Temp

[ ] [ ] [ ] [ ] [ ] NTU Turbidity

FIELD COMMENTS

Sample Appearance: _____
Weather Conditions: _____
Other: _____

Pace Incorporated
New Jersey Laboratory
20x Raritan Center Parkway
P.O. Box 7808
Edison, NJ 08818-7808

FILTERING: Use Chain of Custody (CC1) to indicate which bottles were filtered

Sampler: _____ (Print) Employer: _____

I certify that sampling procedures were in accordance with applicable EPA state and corporate protocols.

_____ (Date) _____ (Signature)

Figure 7.4

PACE/New Jersey Laboratory
LOGIN CHAIN OF CUSTODY REPORT (ln01)
Aug 29 1994, 01:46 pm

Login Number: LDCZ9
Receive Date Range: 29-AUG-94 thru 29-AUG-94
Account: A-150 ETC QA
Project: NC CERTIFICATION PE'S

Laboratory SampleNumber		Client SDG SampleNumber	Collect Date	Receive Date	PR	Due Date
DCZ9-05		NCTOC-D	29-AUG-94	29-AUG-94		16-SEP-94
SPECCV--REMEDIAL PE FOR NC. FOLLOW INSTRUCTIONC (SEE SUPERVISOR) DATA DUE TO QA BY 9-16 PREPARE AND ANALYZE IN DUPLICATE SPECRP--NO TECH REPORT						
Aqueous	S	415.1/TOC4	ETED Hold:26-SEP	403276		
No Matrix	S	RPTVAL	ETED Hold:29-AUG	403276		
Aqueous	S	SPECCV	ETED Hold:05-SEP	403276		
No Matrix	S	SPECRP	ETED Hold:26-SEP	403276		

Date:

CLIENT CHAIN OF CUSTODY RECORD

Shipping Containers Received by: ETC CORPORATION
2c RARITAN CENTER PARKWAY
EDISON, NJ 08818-7808

Date: _____ Time: _____

Number of ETC Shuttles/coolers/other shipping contain_____

Client: _____ _____

_____

Comments: _____

_____

_____

Relinquished by: _____

_____

Received by: _____

_____

Provide copy of this transfer record to client (or designate).
Retain record with shipping container(s) prior to sample log-in.
Maintain original record in Chain of Custody file.

1/91, 2/91 Rev

Figure 7.6

ETC

CHAIN OF CUSTODY

COMPANY: _____

ADDRESS: _____

_____

Attn: _____

SAMPLE DESCRIPTION:

CUSTOMER ID	DESCRIPTION	ETC #

Pace Incorporated
New Jersey Laboratory
284 Raritan Center Parkway
P. O. Box 7608
Edison, NJ 08818-7608

Sample(s) relinquished by: _____

Date: _____ _____ Time: _____

Sample(s) received by: _____

Date: _____ Time: _____

Shuttle Number: _____ Seal Number: _____ Temp: _____

Request for Subcontract Analysis and Sample Chain-of-Custody

Name of Subcontract Laboratory: _____

Loglink(s): _____ **Workgroup:** _____

Matrix: _____ **Sampling date(s):** _____

ETC Sample ID Numbers: _____ _____

_____

_____

Turnaround is 14 days unless otherwise indicated. _____
(If deadline can not be met, contact **ETC Subcontract Department - (908)225-6764**
Send report and invoice to **ETC Corp. P.O.Box 7808, Edison, NJ 08818-7808**
Attention: Subcontract Department

The following analyses are requested:

_____ ACID	_____ CY/T	_____ SOLIDS/SETL
_____ ALKA	_____ FLUORIDE	_____ SOLIDS/TS
_____ NH3	_____ FORM/UV	_____ SOLIDS/TV
_____ NH3/D	_____ HARD	_____ SO4
_____ ASBESTOS/EM	_____ NO3	_____ SULFIDE
_____ ASBESTOS/LM	_____ NO2	_____ SO3
_____ BICARB	_____ N2/ORG	_____ SURFAC
_____ BOD	_____ N2/TK	_____ TOX
_____ BROM	_____ ODOR	_____ TS
_____ BTU	_____ O+G/GRAV	_____ TV
_____ CARB	_____ O+G/IR	_____ TVS
_____ CA/CACO3	_____ PETHY/GRAV	_____ TOC
_____ CHLOR	_____ PETHY/IR	_____ DW1/ABC
_____ CL2/T	_____ PHENOLS	_____ DW1/RA226
_____ CL2/D	_____ PHENOLS/DL	_____ DW1/RA228
_____ CL2/R	_____ PO4/T	_____ AP9/SULFIDE
_____ COD	_____ PO4/ORG	_____ AP9/CY
_____ COLI/F	_____ PO4/ORT	_____ COMPCV
_____ COLI/T	_____ PHOS	_____ FILT
_____ COLOR/S	_____ CY/REACT	_____ HOMO
_____ COLOR/A	_____ SULFIDE/REACT	_____ ECRA/PETHYIR
_____ CR(+6)	_____ SOLIDS/T	
_____ CY(CHLOR)	_____ SOLIDS/TD	

Others: _____

_____

_____

Chain-of-custody (complete appropriate section)

Option A: Courier pickup at ETC
Sample(s) relinquished by ETC: _____
Time: _____ Date: _____
Sample(s) received by: _____
Time: _____ Date: _____

Option B: Sending sample from ETC
Shuttle sealed at ETC by: _____
Date: _____ Time: _____ Seal Number: _____
Shuttle opened by: _____
Date: _____ Time: _____ Seal Number: _____
Seal intact? yes / no Shuttle contents in good condition? yes / no

Figure 7.8

PACE
INCORPORATED
INTERNAL CHAIN OF CUSTODY

Laboratory: PACE Location: Edison, NJ

Client:	*Analytical Parameter/Fraction:*

Sample No.	Batch No.	Sample No.	Batch No.

Date/Time	Relinquished by	Received by	Purpose of Change of Custody
	Printed Name	Printed Name	
	Signature	Signature	
	Printed Name	Printed Name	
	Signature	Signature	
	Printed Name	Printed Name	
	Signature	Signature	
	Printed Name	Printed Name	
	Signature	Signature	
	Printed Name	Printed Name	
	Signature	Signature	
	Printed Name	Printed Name	
	Signature	Signature	
	Printed Name	Printed Name	
	Signature	Signature	
	Printed Name	Printed Name	
	Signature	Signature	
	Printed Name	Printed Name	
	Signature	Signature	

Figure 7.9

PACE/New Jersey Laboratory — LAB CHRONICLE: Sample Preparation (wk02s) — Sep 13 1994

WORK GROUP: Q1 — SCHEDULER: — CREATED : 13-SEP-94 — DUE:

Sample	SDG	Locatn	Ver Sur	Sample Amt. ml	gm	Moisture %	Dry Wt. gm	EXT VOL (ml)		VB HOLD	VB DUE	HC DUE	REPEAT Orig	QS	No.	M	ANALYSIS
										Verify Spike							
QS																	
QS		s1															
		ms1															
		msd1															
QS																	
QS		s2															
		ms2															
		msd2															

Comments:

PROCEDURE:

	C1 SIGNATURE	C1 DATE	C2 SIGNATURE	C2 DATE
__ CONTINUOUS				
__ SEP. FUNNEL				
__ SONICATION				
__ SOXHLET				
__ DILUTION				
__ DERIVATION				
__ A/B PART.				
__ FLORISIL CART.				
__ ALUMINA				
__ SULF/COPPER				
__ GPC				
__ CONC.				

SPIKE	VOL ml	CONC. ug/ml	CONC. mg/ml	LOT NUMBER
SURROGATES				

SOLVENT	LOT NUMBER

REAGENT	LOT NUMBER
Sodium Sulfate	
Florisil	

Matrix: __ Water __ Soil __ Complex __ Organic Liq.

ESD: _____ ECD: _____

SUPERVISOR: _____ DATE: _____

PACE
INCORPORATED

DIOXIN LABORATORY CHRONICAL

SAMPLE MATRIX: 1 – WATER 2 – SOIL 3 – COMPLEX 4 – OIL

HOLD DATE: **DUE DATE:**

ETC SAMPLE NUMBER	CLIENT ID	ETC LOGLINK	A – TYPE	ORIGINAL BATCH	SAMPLE WT. (g) VOL. (ml)	FINAL VOL. ul
A						
B						
S						
MS						
MSD						

PROCEDURES	PERFORMED BY	DATE	LOT NUMBER	CONC ug/ml	VOLUME ul	
WEIGHT OUT						
Sodium Sulfate						
Spike ISTD / SURR						
NATIVE						
JAR SHAKE						
SOXLET						
SEP FUNNEL						
MSSG Silica Gel(_____ X)						UPDATES
ACID WASH(_____X)						QS
BWA (Alumina)						WIP
AX21 (Carbon)						DONE
PACKAGING						END

Solvent	Lot No.

ANALYST'S SIGNATURE:_____ DATE:_____

SUPERVISOR'S SIGNATURE:_____ DATE:_____

TCLP PREP LOG (METHOD 1311) SEMI–VOLATILE ORGANICS AND METALS

QC BATCH # _____ **DUE DATE:** _____

Sample ID	LOG Link	Hold Time	Pretest 1 pH	Pretest 2 Ph	Extr. Fluid	Sample Wt. (g)	Initial Filtrate	Total Volume	Leachate pH	Leach before tumbling	Fraction

SET UP: _____

FILTERING: _____

SUPERVISOR: _____

EXTRACTION FLUID LOT #: _____

COMMENTS:

See attatched logins and Spec sheets for special requirements and instructions.

_____

_____

_____

Start Time: _____

Lab Temp: _____

Tumbler RPM: _____

Stop Time: _____

TCLP PREP LOG (METHOD 1311) VOLATILE ORGANICS (ZHE)

QC BATCH # _____

SAMPLE ID	LOG LINK	TCLP HOLD DATE	SAMPLE DUE DATE	SAMPLE WGT	INITIAL FILTRATE	TOTAL VOL	FRACTION

SET UP: _____ START TIME: _____

'LTERING: _____ LAB TEMP: _____

.JPERVISOR: _____ TUMBLER RPM: _____

EXTRACTION FLUID LOT #: _____ STOP TIME: _____

COMMENTS:

See attatched logins and Spec sheets for special requirements and instructions.

BDI811 – NJREDDELIV

Figure 7.10

PACE/NJ

LABORATORY CHRONICLE: GC/MS DEPARTMENT
SEMI-VOLATILE ANALYSIS

Page _____ of _____

Date: _____ Instr. _____
GC Column: _____
Analyst: _____
A-Type: _____
Tune File: _____
Seq. File: _____
Method File: _____
ID File: _____
CB File: _____
Reviewed By/Date: _____
Batch #s: _____
_____

Standard	Conc ppm	Lot No.	Lot Val
Solvent	—	Lot No.	—
	—		—

Standards Updated

Date: _____ By: _____

Tape #: _____ Inj. _____ ul

R	Name	Data File	ALS #	Dil	Time	Comments	+ P

R: Redo / P: Aroclor Search / + : Plus Search

PACE/NJ

LABORATORY CHRONICLE: GC/MS DEPARTMENT
VOLATILE ANALYSIS

Date: _____ Instr._____
GC Column: _____

Batch #: _____

Water/Soil: _____
A-Type: _____
Tune File: _____
Seq. File: _____
Method File: _____
ID File: _____
CB File: _____

Analyst: _____

Standards Updated:

Date: _____
By: _____

Reviewed By: _____
Date: _____

Tape # _____

Standard	Conc ppm	Lot No.	Lot Vol.
Solvent	----	Lot No.	----
	----		----

R	pH < 2 Y/N	Name	Data File	Init. vol/wt (ml/g)	M	ALS #	Dil	Inj. Time	Comments	+

R: Redo / M: Methanol Dispersion / + : Plus Search

PACE/NJ

Figure 7.11

LABORATORY CHRONICLE: GC DEPARTMENT
SEMI-VOLATILE ANALYSIS

Date: _____ Instr. _____
A-Type: _____
Column: _____
Col. Lot: _____
Calib File: _____
Seq. File: _____
Method File: _____
ID File: _____
Config. File: _____
Analyst: _____
Reviewed By/Date: _____
Batch #s: _____

Standard	Conc ppm	Lot No.	Standard	Conc ppm	Lot No.
			Solvent	-	Lot No.
				-	

Standards Updated

Date: _____ By _____

Tape #: _____ Inj. _____ ul

R	Name	Data File Detec. 1	Data File Detec. 2	init. ml/g	ALS #	Dil	Comments

R: Redo / P: Aroclor Search / + : Plus Search

PACE/NJ

LABORATORY CHRONICLE: GC DEPARTMENT
VOLATILE ANALYSIS

Date: _____ Instr._____
GC Column: _____

Batch #: _____

Water/Soil: _____
A-Type: _____
Tune File: _____
Seq. File: _____
Method File: _____
ID File: _____
CB File: _____

Analyst: _____

Standards Updated:

Date: _____
By: _____

Reviewed By: _____
Date: _____

Tape # _____

Standard	Conc ppm	Lot No.	Lot Vol.
Solvent	----	Lot No.	----
	----		----

R	pH < 2 Y/N	Name	Data File	Init. vol/wt (ml/g)	M	ALS #	Dil	Inj. Time	Comments	+

R: Redo / M: Methanol Dispersion / + : Plus Search

Figure 7.12

PACE/NJ

LABORATORY CHRONICLE: LC DEPARTMENT

Page _____ of _____

Date: _____ Instr._____

Analyst: _____
A-Type: _____
Seq. File: _____
Method File: _____
ID File: _____
CB File: _____
Reviewed By/Date: _____
Batch #s: _____
_____

Standard	Conc ppm	Lot No.
Solvent	—	Lot No. —
	—	—

Standards Updated

Date: _____ By:_____

Tape #: _____ Inj. _____ ul

R	Name	Data File	ALS #	Dil	Comments

R: Redo / P: Aroclor Search / + : Plus Search

Figure 7.13

PACE/NJ

SAMPLE PREPARATION LOG - METALS

BATCH # _____ MATRIX _____

Sample ID	ICP Initial	ICP Final	Furnace Initial	Furnace Final	Mercury Initial	Mercury Final
(LCS) Control						
(PB) Q -1						
(BS) Q -A1						

Matrix Spike and Duplicate Information

Sample ID							
	-1						
(MS)	A1						
(D)	-2						

Analyst :						
Date:						
	HNO3 Lot # :		HNO3 Lot # :		HNO3 Lot # :	
	HCl Lot # :		H2O2 Lot # :		H2SO4 Lot # :	
	H2O2 Lot # :				HCl Lot # :	
					KMnO4 Lot # :	
					K2S2O8 Lot # :	

Figure 7.14

PACE/NJ

Page _____

MERCURY LABORATORY CHRONICLE

	Sample ID	Dilution		Sample ID	Dilution
1			23		
2			24		
			25		
			26		
5			27		
6			28		
7			29		
8			30		
9			31		
10					
11					
12			34		
13			35		
			37		
16			38		
17			39		
18			40		
19			41		
20			42		
21			43		
22			44		

Folder # : _____ **Batches :** _____

Analyst: _____ **Da** ____/____/____

Ca' ion Standard Lot # : _____ _____

C...ation Check Lot # : _____ _____

Stannous Chloride Lot # : _____

Hydroxylamine Hydrochloride Lot # : _____

PACE/NJ

LABORATORY CHRONICLE - GFAA

Date: ____/____/____ Instrument FN / : _____ Analyst: _____

QC Batch (s): _____ Matrix: _____

Calibration Standards Lot # : _____

Calibration Check Standard Lot # : _____

	Element	Position	File
1			
2			
3			
4			
5			

POS	SAMPLE	LOT #	POS	SAMPLE	DIL	POS	SAMPLE	DIL
1	SXS00		15			29		
2	SASTDS10		16			30		
3	STSTDS20		17			31		
4	SCSTDS40		18			32		
5	SDSTDS60		19			33		
6	CSFICV		20			34		
7	CBICB		21			35		
8			22			36		
9			23			37		
10			24			38	NH4H2PO4	
11			25			39	NI(NO3)3	
12			26			40	PSTSPK SOLN	
13			27			ALT	H2SO4 5%	
14			28					

PACE/NJ

LAB CHRONICLE - ICP

Page _____ of _____

Date: _____ Instrument: _____ Analyst: _____

QC Batch (s): _____ Matrix: _____

Sequence File: _____

Calibration Standard Lot # : _____

Calibration Check Lot # : _____

CRI Lot # : _____

ICSA Lot # : _____

ICSAB Lot # : _____

POS	SAMPLE	DIL/ELE	POS	SAMPLE	DIL/ELE	P(	SAMPLE	DIL/ELE

Figure 7.15

PACE/NJ

CHLORIDE

N-Batch: _____ Method Ref: EPA 325.2, SW-846 9251 Page _____of _____

QC-Batch: _____ Colorimetric/Automated Feri Cyanide) Date: _____

Verified: _____ MDL: 1.0 mg/l Time: _____

Instrument: _____ Matrix: Aqueous Analyst: _____

Seq #	ETC Job #	Analyzed mg/l	Dilution Factor	Reported mg/l	Comments Calculations

Traceability

Std Lot #: _____ External Ref #: _____ Blank Spike Lot #: _____

Color Reagent #: _____

TABLE 8.1 ROUTINE INSTRUMENT CALIBRATION SUMMARY

INSTRUMENT	MODEL	METHODS	STANDARD RANGE	PROCEDURE	FREQUENCY
Wet Chemistry					
Spectrophotometer	Technicon AA II Technicon GTPC (Auto Analyzers)	Phenolics - 420.2/9066 Chloride - 325.2/9251 Cyanide - 335.2/9012 Cyanide - OLM CLP Nitrate - [illegible] Nitrite - 353.2 Fluoride - 4500-F-E	0.05 - 0.30 mg/L 5.0 - 200 mg/L 0.10 - 0.50 mg/L - 200 mg/L 0.10 - 2.0 mg/L 0.05 - 1.0 mg/L 0.10 - 2.0 mg/L	5 point calib. 5 point calib. 5 point calib. 5 point calib. 5 point calib. 5 point calib. 5 point calib.	per batch
	Hach DR3000	COD - Hach 8000, 1979	10.0	5 point calib.	per batch
Carbon Analyzer	Dohrmann DC80 (w/ soil furnace)	TOC - 415.1/9060	10.	Single point; 5 pt verif.	per batch; per batch
Infrared Spectrophometer	Perkin Elmer 1310	Petroleum Hydrocarbon - 418.1	0.5 - 40 mg/L	5 point calib.; 2 point verification	quarterly; per batch
pH Meter	Orion 501, 701	pH - 150.1/9040	4 - 10 units	3 point calib.	per batch
Conductivity Meter	YSI #32	Specific Conductance 120.1/9050	0.01 NKCL @ 1413 umhos/cm	Standard	per batch
Turbidity Meter	Hach [illegible]	Turbidity - 180.1	1.8 - 1[illegible] NTU	3 point calib. (Formazin); 3 point calib.	Quarterly; per batch
Turbidity Meter	Hach X/R	Surface 5.4	5.0 - 150 mg/L	5 point calib.	per batch
Seta-Flash Unit	ERDCO DISF	Flash Point - 1020	27.2° C	single point p-Xylene	per batch
Tox Analyzer	Dohrmann DX20A Mitsubishi TOX-10	TOX - 9020	1.0 ug Cl	Cal. check Std	per batch; 1/8 pyrolysis
Metals					
Mercury Cold Vapor Atomic Absorption Spectrophotometer	Fisher Hg-3 Analyzer	CVAA Hg - 245.1/7470,74 USEPA CLP	0.[illegible] ug/L	6 point calib.; calibration check	Initial; Every 10 samples
Graphite Furnace Atomic Absorption Spectrophotometer	Perkin Elmer 5100 with Zeeman background correction	GFAA As - 206.2/7060/CLP Se - 270.2/7740/CLP Tl - 279.2/7841/CLP Pb - 239.2/7421/CLP Cd - 213.2/7131 Cr - 218.2/7191 Sb - 204.2/7041	By Element (ug/L) 0 - 40 0 - 40 0 - 40 0 - 40 0 - 6 0 - 12 0 - 50	4 point calib.; calibration check	Initial; Every 10 samples

TABLE 8.1 CONTINUED

INSTRUMENT	MODEL	METHODS	STANDARD RANGE	PROCEDURE	FREQUENCY
Metals continued					
ICP Atomic Emission Spectrophotometer	Jarrell Ash ICP-61E ICP-61	ICPAE Ag - 200.7/6010/CLP Al - 200.7/6010/CLP As - 200.7/6010/CLP B - 200.7/6010 Ba - 200.7/6010/CLP Be - 200.7/6010/CLP Ca - 200.7/6010/CLP Cd - 200.7/6010/CLP Co - 200.7/6010/CLP Cr - 200.7/6010/CLP Cu - 200.7/6010/CLP Fe - 200.7/6010/CLP Mg - 200.7/6010/CLP Mn - 200.7/6010/CLP Mo - 200.7/6010 Na - 200.7/6010/CLP Ni - 200.7/6010/CLP K - 200.7/6010/CLP Pb - 200.7/6010/CLP Sb - 200.7/6010/CLP Se - 200.7/6010/CLP Sn - 200.7/6010 Sr - 200.7/6010 Ti - 200.7/6010 V - 200.7/6010/CLP Zn - 200.7/6010/CLP	By Element (mg/L) 0 - 0.2 0 - 5.0 0 - 1.0 0 - 1.0 0 - 2.0 0 - 0.2 0 - 20 0 - 0.2 0 - 0.4 0 - 0.6 0 - 0.6 0 - 2.0 0 - 20 0 - 0.6 0 - 0.5 0 - 100 0 - 0.6 0 - 20 0 - 1.0 0 - 1.0 0 - 2.0 0 - 2.0 0 - 1.0 0 - 1.0 0 - 0.6 0 - 1.0	4 point calib.; calibration check	Initial; Every 10 samples
Organics					
Tracor Gas Chromatograph	540	Purgeable Halocarbons 601	By Compound (ug/L) 0.50 - 25 1.0 - 25	5 point calib.; calibration check	Initial; After 10 injections
		Purgeable Halocarbons 8010	1.0 - 100 ug/L	5 point calib.; calibration check	Initial; After 10 injections
		Purgeable Aromatics 602/8020	1.0 - 100 ug/L	5 point calib.; calibration check	Initial; After 10 injections
Hewlett Packard Gas Chromatograph	5890A 5880A	Purgeable Aromatics 602/8020	1.0 - 100 ug/L	5 point calib.; calibration check	Initial; After 10 injections
		Organochlorine Pesticides/PCBs 608	By Compound (ug/ml) 0.0005 - 0.20 0.0010 - 0.40 0.0050 - 2.0 0.010 - 4.0 0.020 - 8.0	5 point calib.; calibration check	Initial; After 10 injections

TABLE 8.1 CONTINUED

INSTRUMENT	MODEL	METHODS	STANDARD RANGE	PROCEDURE	FREQUENCY
Organics continued					
		Organochlorine Pesticides/PCBs 8080	By Compound (ug/ml) 0.005 - 0.20 0.010 - 0.40 0.050 - 2.0 0.10 - 4.0 0.20 - 8.0	5 point calib.; calibration check	Initial; After 10 injections
		Organophosphorus Pesticides - 8140	By Compound (ug/ml) 0.10 - 1.60 0.05 - 0.80 0.025 - 4.0	5 point calib.; calibration check	Initial; After 10 injections
		Chlorinated Herbicides - 8150	By Compound (ug/ml) 0.50 - 10 0.10 - 2.0	5 point calib.; calibration check	Initial; After 10 injections
		Organochlorine Pesticides and PCBs USEPA CLP	By Compound (pg) 5 - 80 10 - 160 50 - 800 100, 200, 500	3 point calib; calibration check	Initial, Every 12 hours
Hewlett Packard Gas Chromatograph/ Mass Spectrometer	5995 5995A 5988 5970A 5890A	Volatiles, purge & trap 624	4 - 200 ug/L	5 point calib.; calibration check	Initial; Every 12 hours
		Volatiles, purge & trap 8240	20 - 200 ug/L	5 point calib.; calibration check	Initial; Every 12 hours
		Volatiles, heated purge & trap - 8240	50 ug/L	5 point calib.; calibration check	Initial; Every 12 hours
		Volatiles, purge & trap 524.2	1 - 20 ug/l	5 point calib.; calibration check	Initial; Every 8 hours
		Semivolatiles (extractables) - 625	By Compound (ug/ml) 10 - 160 20 - 160 80 - 160	3/5 point calib.; calibration check	Initial; Every 12 hours
		Semivolatiles (extractables) - 8270	20 - 160 ug/ml	5 point calib.; calibration check	Initial; Every 12 hours
		PCDD, PCDF, 2,3,7,8-TCDD - 8280	20 - 500 ng	5 point calib.; calibration check	Initial; Every 12 hours
		Volatiles, purge & trap USEPA CLP	500 - 1000 ng	5 point calib.; calibration check	Initial; Every 12 hours

TABLE 8.1 CONTINUED

INSTRUMENT	MODEL	METHODS	STANDARD RANGE	PROCEDURE	FREQUENCY
Organics continued					
		Semi-Volatiles (extractables) - USEPA CLP	20 - 160 ng	5 point calib.; calibration check	Initial; Every 12 hours
Hewlett Packard High Performance Liquid Chromatography	1090 1090A	Polynuclear Aromatic Hydrocarbons - 8310	By Compound 0.25 - 5 0.5 - 10 2.5 - 50 5.0 - 100; or 0.025 - 1.0 0.05 - 2.0 0.25 - 10.0 0.5 - 20.0	5 point calib.; calibration check	Initial; Daily verification

Figure 10.1

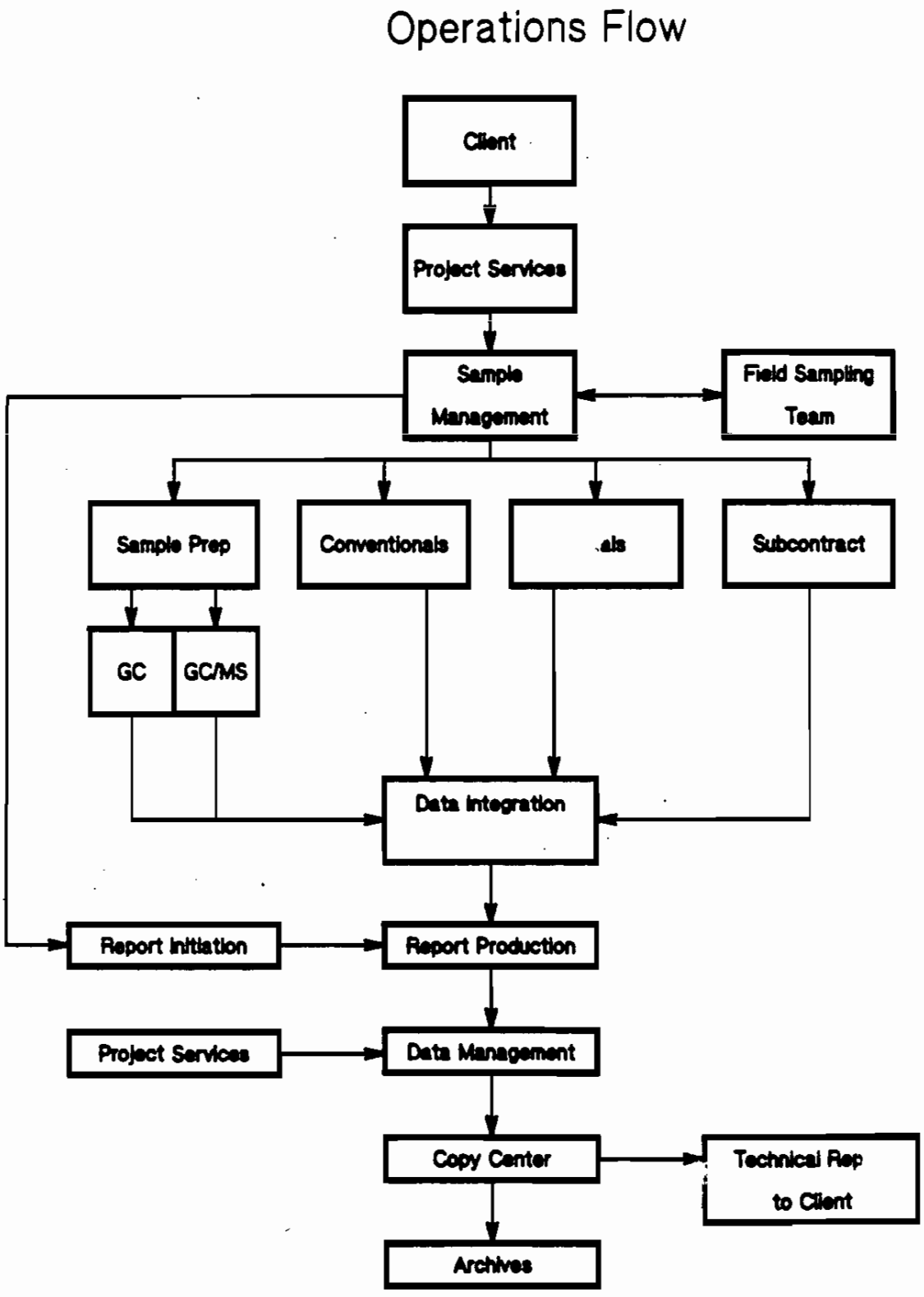

Operations Flow
Client
Project Services
Sample Management
Field Sampling Team
Sample Prep
Conventionals
als
Subcontract
GC
GC/MS
Data Integration
Report Initiation
Report Production
Project Services
Data Management
Copy Center
Technical Rep to Client
Archives

Table 12.1 State Certification Summary - September 1994

State Agency/Certification Type	Certification ID No.
C rnia Department Health Services/ Hazardous Waste Fields of Testing	1814
Connecticut Department of Health Services/ Potable Water, Non-potable Water, Soils	PH-0511
Kansas Department of Health and Environment/ Non-potable Water, Hazardous Waste	E148, E1122
Massachusetts Department of Environmental Protection/ Potable Water, Non-potable Water	M-NJ136
New Hampshire Department of Environmental Services/ Non-potable Water	202693-E
New Jersey Department of Environmental Protection/ Potable Water, Non-potable Water	12941*
New York Department of Public Health/ Potable Water, Non-potable Water, Hazardous Waste	5
North Carolina Department of the Environment/ Non-potable Water	326
Oklahoma Department of Environmental Quality/ Non-potable Water	8703
Pennsylvania Departme f Environmental Resources/ Potable Water	68-323
South Carolina Department of Health d Environmental Control/ Non-potable Water, Solid and Hazardous Waste	94002
Tennessee Department of Health and Environment/ Potable Water	02915
Utah Department of Health/ Potable Water, Non-potable Water, H rdous Waste	E-91
Virginia Department of General Servi / Potable Water	00113
Wisconsin Department of Natural Resources/ Non-potable Water	999464070

List of Actual Certified Tests/Parameters Available Upon Reque

* The N EP ID will becor e #12005 1 is current as of this document's approval date.

Figure 15.1

ETC

ROUTINE CC -CTIVE ACT -OG

Batch #_____Analysis_____ _Matrix_____

Preparation by:_____

Ana d by:_____

Data Reviewer:_____

DATE	ISSUE/RESOLUTION

Pace Incorporated
New Jersey Laboratory
284 [illegible]
P
Ed
'308
[illegible]

Figure 15.2

ETC

INVESTIGATION/CORRECTION LOG

Log #: ____________________

QA Receipt Date: ____________________

Originator: ____ Date: ______________

Requested by: ETC____ Client_____ Other__________ Resolution required by (date):__________

Sample Information (Complete only if sample specific):

Company: ____

Contact: ______________________ Phone: ( ) ______________ Fax: ( ) ______________

ETC Sample #s: ____

Client ID #s: ____

Loglink: ____

	Yes	No	Circle One	Completed by	Date
Client to be contacted?					
Corrected report(s) to be issued?			Mail/FedEx		
Letter to be issues?			Mail/Fax		
Other: ______________					

Description: ____

Identify Investigation Responsibility:

QA _____ Mktg _____ PM _____ Ops (Specify Dept): ______________

Pace Incorporated
New Jersey Laboratory
384 Raritan Center Parkway
P. O. Box 7...
Edison, N... ...7808

Investigation: ____

(Attach pertinent documentation)

Performed by: ______________________ Date: ______________

QA Review/Notification o ginator:

Name: ______________________ Date: ______________

_____ No Actio

_____ Simple Corrective Action

Description: ____

_____ Complex Corrective Action

Description: ____

Assigned to: ______________________ Date: ______________

Completed by: ______________________ Date: ______________

(Includes - documentation, implementation, training and verification)

Attach all necessary documentation prior to returning to QA.

Verified by Area Supervisor: ______________ Date: ______________

Monitored by QA: ______________________ Date: ______________